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170608



REMOVAL SUPPORT TEAM
EPA CONTRACT 68-W-00-113

April 26, 2005

Mr. Nick Magriples On-Scene Coordinator
U.S. Environmental Protection Agency, Region II
Removal Action Branch
2890 Woodbridge Avenue
Edison, NJ 08837

EPA CONTRACT NO: 68-W-00-113

TDD NO: 02-05-04-0005

DOCUMENT CONTROL NO: RST-02-F-01821

SUBJECT: QUALITY ASSURANCE PROJECT PLAN

Matteo Iron and Metal Site, West Deptford, Gloucester County, New Jersey

Dear Mr. Magriples:

Enclosed please find the multimedia sampling Quality Assurance Project Plan for the Matteo Iron and Metal Site, 1708 Route 130, West Deptford Township, Gloucester County, New Jersey.

If you have any questions, please do not hesitate to call me at (732) 225-6116, extension 230.

Sincerely,

WESTON Solutions, Inc.

Aaron Levy
Site Project Manager

Enclosure

cc: TDD File: 02-05-04-0005

S. Sumbaly, QAO

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In Association with Scientific and Environmental Associates, Inc.
Innovative Technological Solutions, Inc., and TerranearPMC



SITE QUALITY ASSURANCE PROJECT PLAN

Matteo Iron and Metal Site
1708 Route 130
West Deptford Township, Gloucester County, New Jersey

DCN #: RST-02-F-01821
TDD #: 02-05-04-0005
EPA Contract No.: 68-W-00-113

Prepared by:

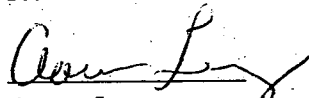
Removal Support Team
WESTON Solutions Inc.
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Edison, New Jersey 08837

Prepared for:

U.S. Environmental Protection Agency
Region II - Removal Action Branch
Edison, New Jersey 08837

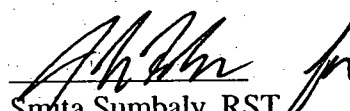
Approved by:

RST


Aaron Levy
Site Project Manager

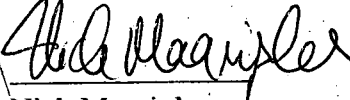
Date: 4/27/05

RST


Smita Sumbaly, RST
Quality Assurance Officer

Date: 4/27/05

EPA


Nick Magriples
On-Scene Coordinator

Date: 5/10/05

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QA REPORTS TO MANAGEMENT
PREVENTIVE MAINTENANCE PROCEDURES AND SCHEDULES
RECORDS MANAGEMENT SYSTEM
LOGBOOK PROGRAM
QUALITY-RELATED DOCUMENTS
INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

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ATTACHMENT B: EPA/ERT SOP No. 2012 - Soil Sampling
EPA/ERT SOP No. 2009 - Drum Sampling
EPA/ERT SOP No. 2011 - Chip, Wipe, Sweep Sampling
EPA/REAC SOP # 1700 Niton XL722S Field Portable X-Ray Fluorescence
Operating Procedure (Draft)

ATTACHMENT C: Drum Inventory and Field Testing Log
Drum Location Log
Air Monitoring Work Sheet

ATTACHMENT D: PCB Field Screening Procedure - SW 846 Method 9078

1.0 INTRODUCTION

Presented herein is the Site Quality Assurance Project Plan (QAPP) for the sampling event to be conducted at the Matteo Iron and Metal Site (Site) by the Region II Removal Support Team (RST). The site QAPP has been developed at the request of the United States Environmental Protection Agency (EPA) in accordance with the RST generic Quality Assurance Project Plan (QAPP).

This plan is based on information currently available and may be modified on-site in light of field screening results and other acquired information. All deviations from the QAPP will be noted in the Sampling Trip Report.

2.0 PROJECT DESCRIPTION

RST is tasked to conduct an Integrated Assessment of the Matteo Iron and Metal Facility, 1708 Route 130, West Deptford Township, New Jersey (see Attachment A - Site Location map). The site is approximately 80 acres in size and consists of a landfill and a recycling facility in the northeast portion. It is situated near the confluence of Hessian Run and Woodbury Creek, approximately one mile east of the Delaware River. In the past, batteries were recycled at the Site using both crushing and burning operations. Wire was also burned for the recovery of metal. Early Responsible Party and DEP investigations identified PCBs and high levels of lead and cadmium in Site soils and lead in a Site potable well. A subsequent EPA investigation estimated the volume of landfilled battery casings on Site to be approximately 235,000 ft³. Lead was also found in the sediments of the Hessian Run and in the marsh areas adjacent to the battery casing disposal area.

EPA Region II has tasked RST and the Site Assessment Team (SAT) to conduct air monitoring, multimedia sampling of drums and soils, and field screening of soils for PCBs and lead at the Matteo Iron and Metal Site. RST will maintain the site logbook to document site activities, prepare a site-specific Health & Safety, QAPP, and Trip Report for EPA TM approval, collect samples, characterize drum samples, provide lab analysis and data validation services, and provide photo-documentation. SAT will create a sampling grid and perform the field screening of soils for lead and PCBs using samples collected from locations on-site and in the trailer park adjacent to the site.

3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The EPA On-Scene Coordinator (OSC), Nick Magriples, will provide overall direction to the RST staff concerning project sampling needs, objectives, and schedule. The RST Site Project Manager (SPM), Aaron Levy, will be the primary point of contact with the OSC. The SPM is responsible for the development and completion of the Sampling QA/QC Plan, project team organization, and supervision of all project tasks, including reporting and deliverables. The RST QC Coordinator, Aaron Levy will be responsible for ensuring field adherence to the RST Sampling QA/QC Plan and recording of any

deviations. The RST Quality Assurance Officer (QAO), Smita Sumbaly, will be the primary project team site contact with the subcontracted laboratory, if necessary.

The EPA Work Assignment Manager (WAM), Kristin Dobinson, will provide overall direction to the SAT staff concerning project sampling needs, objectives, and schedule. The SAT Site Project Manager (SPM), Michele Capriglione, will be the primary point of contact with the WAM. The SPM is responsible for the development and completion of the SAT Sampling QA/QC Plan, project team organization, and supervision of all project tasks, including reporting and deliverables. The SAT Site QC Coordinator, Dan Gaughan will be responsible for ensuring field adherence to the Sampling QA/QC Plan and recording of any deviations.

The following sampling personnel will work on this project:

<u>Personnel</u>	<u>Affiliation</u>	<u>Responsibility</u>
Nick Magriples	Region II, EPA	On-Scene Coordinator
Aaron Levy	Weston-RST	RST Site Project Manager, Sample Management
Michael Mahnkopf	Weston-RST	Drum Sampling, HAZCAT Field Screening, H&S Coordinator
Frank Campbell	Weston-RST	Sampling, Sample Management
John Brennan	Weston-RST	Sampling, Sample Management, QA/QC
Michele Capriglione	Weston-SAT	SAT Site Project Manager, GPS data collection
Heather Carson	Weston-SAT	Field Screening, Niton Portable XRF operator
Jason Standowski	Weston-SAT	Site QC Coordinator, Wetland Delineation
Kathleen Bigelow	Weston-SAT	PCB Field Screening

The following laboratories will provide the following analyses:

<u>Lab Name/Location</u>	<u>Sample Type</u>	<u>Parameters</u>
Ceimic Corporation 10 Dean Knauss Drive Narragansett, RI 02882	Soil	TAL Metals
A4 Scientific 1544 Sawdust Road Suite 505 The Woodlands, TX 77380	Soil	Pest/PCB
TBD	Powdered Solid	Full TCL, TAL Metals, Cyanide, IR

TBD	Drum Waste	TCLP VOC, SVOC, Metals, and Pesticides, Full TCL, TAL Metals, Cyanide, RCRA Characteristics
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A turnaround time of two weeks for written results was requested by the OSC.

4.0 DATA USE OBJECTIVES, QA OBJECTIVES

In addition to the following, the Data Use Objectives, QA Objectives procedure will be conducted in accordance with Sections A7, B2, B4, and B5 of the Region II RST QAPP.

The purpose of the multimedia sampling is to identify the presence or absence of hazardous substances, pollutants or contaminants at the site. This information will be used to supplement existing information in order to determine whether a CERCLA removal action is warranted at the site and to provide data for consideration for placement on the National Priorities List (NPL).

4.1 Data QA Objectives

The overall Quality Assurance (QA) objective for chemical measurement data associated with this sampling event is to provide analytical results that are legally defensible in a court of law. The QA program will incorporate Quality Control (QC) procedures for field sampling, chain of custody, laboratory analyses, and reporting to assure generation of sound analytical results.

The EPA On-Scene Coordinator (OSC) has specified a Level 2 QA objective (QA-2) for soil sample matrices. Soil screening for metals via the XRF has been requested as a QA-2 objective. The drum waste and solid (powdered solid) samples have been specified for a Level 1 QA objective (QA-1), as well as the field screening for PCBs. **Note:** RCRA Characteristics analyses and field characterization of drum waste cannot be performed at QA Level 2.

4.2 QA Objectives

The QA Protocols for a Level 1 QA objective sampling event are applicable to RCRA characteristics and field characterization of drum and soil samples and include:

1. Sample documentation in the form of field logbooks, appropriate field data sheets, and chain of custody records (chain of custody records are optional for field screening locations).
2. Calibration of all monitoring and/or field-portable analytical equipment prior to collection and

analyses of samples with results and/or performance check procedures/methods summarized and documented in a field, personal, and/or instrument log notebook.

3. Field or laboratory determined method detection limits (MDLs) will be recorded along with corresponding analytical sample results, where appropriate.

The QA Protocols for a Level 2 QA objective sampling event are applicable to all sample matrices and include:

1. Sample documentation in the form of field logbooks, appropriate field data sheets, and chain of custody records (chain of custody records are optional for field screening locations).
2. Calibration of all monitoring and/or field-portable analytical equipment prior to collection and analyses of samples with results and/or performance check procedures/methods summarized and documented in a field, personal, and/or instrument log notebook.
3. Field or laboratory determined method detection limits (MDLs) will be recorded along with corresponding analytical sample results, where appropriate.
4. Analytical holding times as determined from the time of sample collection through analysis. These will be documented in the field logbook or by the laboratory in the final data deliverable package.
5. Initial and continuous instrument calibration data.
6. QC blank results (rinsate, trip, method, preparation, instrument, etc.), as applicable.
7. Collection and analysis of blind field duplicate QC sample to provide a quantitative measure of the analytical precision and accuracy, as applicable.
8. Use of the following QC procedure for QC analyses and data validation:

Definitive identification - confirm the identification of analytes on 100% of the "critical" samples, via an EPA-approved method; provide documentation such as gas chromatograms, mass spectra, etc.

Table 1: QUALITY ASSURANCE OBJECTIVES

QA Parameters	Matrix	Intended Use of Data	QA Objective
Full TCLP (minus Herbicides), Full TCL, TAL Metals, Cyanide	Waste	Characterization of material	QA-1
RCRA Characteristics	Waste	Characterization of material	QA-1
XRF Field Screening (Lead)	Soil	Delineate potential lead contamination	QA-2
PCB Field Screening	Soil	Delineate potential PCB contamination	QA-1
PCBs, TAL Metals	Soil	Verify presence or absence of hazardous substances	QA-2
Field Characterization (HAZCAT)	Waste	Field Screening for hazard categorization, selection of laboratory samples and IATA/DOT shipping requirements	QA-1
Full TCL, TAL Metals, CN, IR analysis	Powdered Solid	Characterization of material	QA-1

A Field Sampling Summary is attached in Table 2 and a QA/QC Analysis and Objectives Summary is attached in Table 3. Section 5.1, Sampling Design, provides information on analyses to be performed on the samples collected.

**TABLE 2:
FIELD SAMPLING SUMMARY**

Analytical Parameters	Matrix	Container Size	Preservative	Holding Time¹	Subtotal Samples	Trip Blanks³	Rinsate Blanks²	Duplicate Samples	MS/MSD Samples	Total Field Samples
Full TCLP (minus Herbicides)	Waste	8-oz glass jar, per fraction	ice to 4°C	VOA 14 days to analysis, SemiVOA, Pest/PCB- 7 days for extraction 14 days for analysis	10 ⁴	NR	NR	NR	NR	10 ⁴
RCRA Characteristics	Waste	8-oz glass jar	ice to 4°C	Flash Point - 30 days pH - ASAP NA - all other parameters	10 ⁴	NR	NR	NR	NR	10 ⁴
Lead and Manganese (Field Screening)	Soil	1-gallon Plastic Bag	ice to 4°C	180 days	75	NR	NR	8	NR	83
PCB (Field Screening)	Soil	1-gallon Plastic Bag	ice to 4°C	7 days	75	NR	NR	8	NR	83
PCB	Waste	8-oz glass jar	ice to 4°C	7 days for extraction, 14 days for analysis	10 ⁴	NR	NR	1	1	12 ⁴
PCB	Soil	8-oz glass jar	ice to 4°C	7 days for extraction, 14 days for analysis	23	NR	NR	2	2	27
Cyanide	Waste	8-oz glass jar	ice to 4°C	14 days analysis	10 ⁴	NR	NR	NR	NR	10 ⁴

Analytical Parameters	Matrix	Container Size	Preservative	Holding Time ¹	Subtotal Samples	Trip Blanks ³	Rinsate Blanks ²	Duplicate Samples	MS/MSD Samples	Total Field Samples
TAL Metals	Soil	8-oz glass jar	ice to 4°C	6 months analysis, 28 days mercury analysis	23	NR	NR	2	2	27
Full TCL	Waste	8-oz glass jar	ice to 4°C	VOA 14 days to analysis, SemiVOA, Pest/PCB- 7 days for extraction 14 days for analysis	10 ⁴	NR	NR	NR	NR	10 ⁴
TAL Metals	Waste	8-oz glass jar, per fraction	ice to 4°C	6 months analysis, 28 days mercury analysis	10 ⁴	NR	NR	NR	NR	10 ⁴
TAL Metals, Full TCL, Cyanide, IR analysis	White Powder	8-oz glass jar per fraction	ice to 4°C	Metals - 6 months analysis, 28 days mercury analysis, VOA- 14 days to analysis, SemiVOA, Pest/PCB- 7 days for extraction 14 days for analysis	1	NR	NR	NR	NR	1
Field Screening (HAZCAT)	Waste	8-oz glass jar	ice to 4°C	14 days analysis	10 ⁴	NR	NR	NR	NR	10 ⁴

¹ Holding time from date of sampling.

² Only required if non-dedicated sampling equipment to be used. NR - not required, dedicated sampling equipment to be used.

³ Not required

⁴ Exact number will be based on initial findings and field screening results

TABLE 3
QA/QC ANALYSIS AND OBJECTIVES SUMMARY

Analytical Parameters	Matrix	Analytical Method Reference	QA/QC Quantitation Limits	QA Objective
Full TCLP (minus Herbicides)	Waste	SW 846 Method 1311- extraction; 8270C- BNA; 8260B - VOA ; 8081 - Pesticide; 6010B / 7000 - Metals	As per method	QA-1
Full TCL	Waste	<u>TCL VOA</u> -EPA Method 8260B <u>TCL PCBs</u> - EPA Method 8082 <u>TCL PEST</u> - EPA Method 8081 <u>TCL SVOA</u> - SemiVolatiles-SW846 Method 8270C or CLP OLMO4.2	As per method	QA-1
TAL Metals	Waste	SW846 Method 6010B/7000 or ILMO5.2	As per method	QA-1
RCRA Characteristics	Waste	SW846 Method 1010- Ignitability, Method 9040- corrosivity, Method 9010/9030- Reactivity	As per method	QA-1
Cyanide	Waste	EPA Method 9013/9012A	As per method	QA-1
TAL Metals	Soil	SW 846 Method 6010B/7000 or ILMO 5.2	As per method	QA-2
Field Screening (Lead)	Soil	SOP # 1700 Niton XL722S Field Portable X-Ray Fluorescence Operating Procedure	As per method	QA-2
Field Screening (PCBs)	Soil	SW 846 Method 9078	As per method	QA-1

Analytical Parameters	Matrix	Analytical Method Reference	QA/QC Quantitation Limits	QA Objective
TCL PCBs	Soil	SW 846 Method 8082 or OLMO 4:2	As per method	QA-2
Full TCL	Powder Solid	<u>TCL VOA</u> -EPA Method 8260B <u>TCL PCBs</u> - EPA Method 8082 <u>TCL PEST</u> - EPA Method 8081 <u>TCL SVOA</u> - SemiVolatiles-SW846 Method 8270C or CLP OLMO4.2	As per method	QA-1
TAL Metals	Powder Solid	SW846 Method 6010B/7000 or ILMO5.2	As per method	QA-1
Cyanide	Powder Solid	EPA Method 9013/9012A	As per method	QA-1
IR analysis	Powder Solid	TBD	As per method	QA-1

5.0 APPROACH AND SAMPLING PROCEDURES

In addition to the following, the Approach and Sampling Procedures will be conducted in accordance with Sections B1 and B4 of the Region II RST QAPP.

The following sampling activities will be conducted at the Matteo Iron and Metal Site:

- Soil Sampling
- Drum Sampling
- Drum and container inventory (survey)
- Field characterization of drum waste
- Field screening of soils for lead using XRF.
- Field screening of soils of PCBs using immunoassay tests.

This sampling design is based on information currently available and may be modified on-site in light of field screening results and other acquired information. All sampling activities will be performed by the Region II RST, under the direction of the EPA On-Scene Coordinator (OSC). Any deviations from the sampling plan will be noted in the Sampling Trip Report.

5.1 Sampling Design

During this sampling event, RST will visit the site to inventory all drums on-site and place them onto the drum log sheet (Appendix C). The drum area will be monitored for LEL, Organic Vapors, and radiation. RST will then open and survey each drum. Depending upon the results of this survey, a select number of drums will have samples collected for field screening characterization (HAZCAT) using drum thieves or other devices. Based upon the results of the screening, the drums samples may be composited. The composite samples will then be sent to the laboratory for further analysis.

During this event, four soil samples will be collected from the outside paved area and the street curb area. Soils on top of paved areas will be collected for TAL metals and Polychlorinated Biphenyls (PCBs). Composite soil samples will be collected using plastic disposable scoops, composited in disposable aluminum pans, and placed into the sample jars. If necessary, sweep samples will be taken where the soil layer is not thick enough to use a disposable scoop. In addition, soil samples will be collected from a sampling grid, as described below, on the Site and on the neighboring property, to the west of the Site

from 0-6" for Lead and Polychlorinated Biphenyls (PCBs) field screening. These will be collected using plastic disposable scoops.

As a part of this investigation the Region 2 Site Assessment Team (SAT) was tasked with confirming or denying the presence of PCBs and lead on the Matteo property and at the adjacent property, the Willow Woods Trailer Park. A minimum of 54 samples will be collected on the Matteo property in areas of previously identified PCB contamination, along the boundary of the Matteo property and the adjacent trailer park, and in the trailer park. SAT will screen each sample for the presence of PCBs and lead using PCB immuno-assay kits and an X-Ray Fluorescence machine for PCB and lead detection, respectively. When all of the on site samples are collected and screened, including the boundary samples considered to be on site, 20% of the screened locations will be sampled and sent for Contract Laboratory Program (CLP) analysis for PCBs and TAL Metals. Additionally when all of the trailer park samples are collected and screened including the boundary samples considered to be in the trailer park, 20% of the screened locations will be sampled and sent for CLP analysis for PCBs and TAL Metals.

The on site sample locations were determined by placing a 200-foot grid over areas on the Matteo property where surficial PCB contamination was previously revealed in the Final Remedial Investigation Report (RI) completed in May 2004. There are several locations previously sampled during the 2004 RI from which samples will be collected during this event (Attachment A).

A 100-foot grid will also be placed along the boundary of the trailer park and the Matteo property in order to determine if contamination observed at the Matteo property is affecting the trailer park. A 200-foot grid will be placed over the Willow Woods Trailer Park to confirm or deny the presence of PCBs and lead in the trailer park. Samples collected for screening purposes in the trailer park will be collected from the lawns of the trailer homes.

XRF Field Screening

Field Screening will be completed following SOP 1700 (See Attachment B).

PCB Screening

See Attachment D for PCB field screening procedures (SW 846 Method 9078).

5.2 Schedule of Activities

Proposed Start Date	Activity	End Date
April 27, 2005	Drum Inventory, Air Monitoring, Field Screening, Drum Sampling, Soil Sampling, XRF and PCB Field Screening	April 29, 2005

5.3 Sampling Equipment

Soil samples will be collected with dedicated, disposable plastic scoops and aluminum pans in order to avoid cross-contamination. Drum samples will be collected with drum thieves, coliwasas, or scoops.

5.4 Sample Identification System

Each sample collected by Region II RST will be designated by a code which will identify the site. The code will be a site-specific project tracking number. The code for the Matteo Iron and Metal Site is *MIM*. The media type will follow the numeric code and refer to all matrices or sources. A hyphen will separate the site code and media type. Specific media types are as follows:

- **MIM-SO-** soil
- **MIM-DR-** drum
- **MIM-O-** oil
- **MIM-PS-** powder solid

After the media type, the sequential sample numbers will be listed; sample numbers will be identified as to their location on the site location and/or the location on the \bar{x} and y coordinates of a sampling grid if applicable. A duplicate sample will be identified in the same manner as other samples and will be distinguished and documented in the field logbook.

5.5 Standard Operating Procedures (SOPs)

5.5.1 Sample Documentation

All sample documents will be completed legibly, in ink. Any corrections or revisions will be made by

lining through the incorrect entry and by initialing the error.

FIELD LOGBOOK

The field logbook is essentially a descriptive notebook detailing site activities and observations so that an accurate account of field procedures can be reconstructed in the writer's absence. All entries will be dated and signed by the individuals making the entries, and should include (at a minimum) the following:

1. Site name and project number
2. Name(s) of personnel on site
3. Dates and times of all entries (military time preferred)
4. Descriptions of all site activities, site entry and exit times
5. Noteworthy events and discussions
6. Weather conditions
7. Site observations
8. Sample and sample location identification and description*
9. Subcontractor information and names of on-site personnel
10. Date and time of sample collections, along with chain of custody.
11. Record of photographs
12. Site sketches

* - The description of the sample location will be noted in such a manner as to allow the reader to reproduce the location in the field at a later date.

SAMPLE LABELS

Sample labels will clearly identify the particular sample, and should include the following:

1. Site/project number.
2. Sample identification number.
3. Sample collection date and time.
4. Designation of sample (grab or composite).
5. Sample preservation.

6. Analytical parameters.
7. Name of sampler.

Sample labels will be written in indelible ink and securely affixed to the sample container. Tie-on labels can be used if properly secured.

CUSTODY SEALS

Custody seals demonstrate that a sample container has not been tampered with, or opened. The individual in possession of the sample(s) will sign and date the seal, affixing it in such a manner that the container cannot be opened without breaking the seal. The name of this individual, along with a description of the sample packaging, will be noted in the field logbook.

5.5.2 Sampling SOPs

The following Sampling SOPs will be used for this project:

- EPA/ERT SOP # 2012 - Soil Sampling
- EPA/ERT SOP # 2009 - Drum Sampling
- EPA/ERT SOP # 2011 - Chip, Wipe and Sweep Sampling
- EPA/REAC SOP # 1700- Niton XL722S Field Portable X-Ray Fluorescence Operating Procedure (Draft)

5.5.3 Sample Handling and Shipment

Caps will be secured with custody seals. Bottle labels will contain all required information including the site/project code, sample number, time and date of collection, analyses requested and preservative used. Sealed bottles will be placed in a zip lock plastic bag. The zip lock bag will be placed in large metal or plastic shipping containers. Soil samples will be cooled to 4°C and delivered to the analytical laboratory. All packaging will conform to IATA Transportation regulations for overnight carriers, if applicable.

All sample documents will be sealed in a plastic bag and affixed to the underside of each cooler lid. The lid will be sealed and affixed on at least two sides with custody seals so that any sign of tampering is easily visible.

5.6 Sample Containers

All sample containers will meet the QA/QC specifications in OSWER Directive 9240.0-05A, "Specifications and Guidance for Contaminant Free Sample Containers".

5.7 Disposal of PPE and Contaminated Sampling Materials

All PPE used by RST will be decontaminated on-site and disposed of in appropriate trash receptacle during this site investigation. Investigation derived waste (IDW) from the drum sampling event will be returned to the appropriate drum. SAT personnel will be responsible for the disposal of the IDW generated from the PCB test kits.

6.0 SAMPLE CUSTODY

In addition to the following, the Sample Custody procedure will be conducted in accordance with Section B3 of the Region II RST QAPP.

A chain of custody record will be maintained from the time the sample is taken to its final deposition. Every transfer of custody must be noted and signed for, and a copy of this record kept by each individual who has signed. When samples (or groups of samples) are not under direct control of the individual responsible for them, they must be stored in a locked container sealed with a custody seal. Specific information regarding custody of the samples projected to be collected on the weekend will be noted in the field logbook.

The chain of custody record should include (at minimum) the following:

1. Sample identification number
2. Sample information
3. Sample location
4. Sample date
5. Name(s) and signature(s) of sampler(s)
6. Signature(s) of any individual(s) with custody of samples

A separate chain of custody form must accompany each cooler for each daily shipment. The chain of custody form must address all samples in that cooler, but not address samples in any other cooler. This practice maintains the chain of custody for all samples in case of mis-shipment.

7.0 FIELD INSTRUMENT CALIBRATION AND PREVENTIVE MAINTENANCE

In addition to the following, the Field Instrument and Preventative Maintenance procedure will be conducted in accordance with Section B6 of the Region II RST QAPP.

The sampling team is responsible for assuring that a calibration/maintenance log will be brought into the field and maintained for each measuring device. Each log will include at a minimum, where applicable:

- name of device and/or instrument calibrated
- device/instrument serial and/or ID number
- frequency of calibration
- date of calibration
- results of calibration
- name of person performing the calibration
- identification of the calibrant

Equipment to be used each day will be calibrated prior to the commencement of daily activities.

8.0 ANALYTICAL METHODS

The purpose of the following tests is to classify various unknown containerized waste materials into compatible groups based on their physical and chemical characteristics.

All field compatibility tests will be completed in accordance with the HAZCAT® field identification manual.

1. Air Reactivity - Each sample will be opened and checked for any reaction.
2. Water Reactivity - Each sample will be checked for reactivity by adding nine (9) parts deionized water to one (1) part sample.
3. Water Solubility - Each sample will be checked for water solubility by placing one (1) mL portion of the sample into a small test tube containing nine (9) mL of deionized water. The sample is thoroughly mixed and the following observations will be made:
 1. Sample soluble in water; indicates an inorganic compound or a polar organic

compound;

2. Sample partially soluble in water or forms emulsion in water;
3. Sample insoluble in water and less dense than water;
4. Sample insoluble in water and more dense than water;
5. Sample glimmering in water.

4. Hexane Solubility - Each sample will be checked for hexane solubility by placing one (1) portion of the sample into a small test tube containing nine (9) portions of hexane. The sample is thoroughly mixed and the following observations will be made:

1. Sample soluble in hexane; indicates a possible organic compound;
2. Sample partially soluble in hexane;
3. Sample insoluble in hexane.

5. pH Test - The pH will be measured on aqueous samples and water soluble solid phase samples. The pH will be determined with pHdriion paper. The following observations will be made:

1. Sample with $\text{pH} < \text{or} = 2$; indicates a compound meeting the characteristic of corrosivity;
2. Sample with $\text{pH} > 2, < 12.5$; indicates non-corrosive compound;
3. Sample with $\text{pH} > \text{or} = 12.5$; indicates a compound meeting the characteristic of corrosivity;

If a color change appears that does not match the pH chart, the sample will be diluted with additional water and retested. The dilution will be noted on the drum inventory log.

6. Oxidizer Test - The oxidizer test will be performed on all samples. Potassium iodine-starch test paper will be acidified with one normal hydrochloric acid. The test strip will be touched to the sample. If it turns black or brown, the test is positive.

7. Peroxide Test - The peroxide test will be performed on aqueous and organic phase liquids and hexane and water soluble solid phase samples. Emquant-r peroxide test strips will be used to determine the presence of peroxides. If the test strip changes color, beige to gray or blue, the test is usually positive.

8. Cyanide Test - The cyanide test will be performed on aqueous phase and water soluble

solid phase samples. The sample will be acidified and the vapors will be checked with cyanosmo test paper.

9. Sulfides Test - The sulfides test will be performed on aqueous phase and water soluble solid phase samples. Lead acetate test paper will be moistened with deionized water. The paper is placed over a portion of the sample which will be acidified with 1N hydrochloric acid. If the paper turns black, the test is positive.
10. Chlorinated Solvents Screen - Each organic sample will be checked for the presence of chlorinated organic compounds. A portion of the sample will be placed on clean, 18 gauge copper wire. The sample and the wire will be held in a flame. If it turns the flame green, the test usually indicates the presence of chlorine.
11. Flammability/Ignitability Test - This test will be performed by pouring enough liquid or solid sample into a small dish to cover the bottom of it. A lighted match will be slowly passed under the dish in order to heat the sample. After the sample is sufficiently heated, the match will be placed in the sample. The following observations will be made:
 1. The sample ignites; If the flame is nearly invisible, the sample may be an alcohol. If the flame is bright yellow and emits black smoke, the sample is a pure hydrocarbon compound.
 2. The sample ignites, but sizzles; indicated the sample is an organic compound, but contains water;
 3. The sample does not ignite; indicates sample is an inorganic compound or an organic compound with a high flash point.

Analytical methods to be utilized in the analyses of samples collected during this sampling event are detailed in Table 3.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

In addition to the following, the Data Reduction, Validation, and Reporting procedure will be conducted in accordance with Sections D1, D2, and D3 of the Region II RST QAPP.

9.1 Deliverables

The RST SPM, Aaron Levy, will maintain contact with the EPA OSC, Nick Magriples, to keep him informed about the technical and financial progress of this project. This communication will commence with the issuance of the work assignment and project scoping meeting. Activities under this project will

be reported in status and trip reports and other deliverables (e.g., analytical reports, final reports) described herein. Activities will also be summarized in appropriate format for inclusion in monthly and annual reports.

The following deliverables will be provided under this project:

TRIP REPORT

A trip report will be prepared to provide a detailed accounting of what occurred during each sampling mobilization. The trip report will be prepared within two weeks of the last day of each sampling mobilization. Information will be provided on time of major events, dates, and personnel on site (including affiliations).

MAPS/FIGURES

Maps depicting site layout, contaminant source areas, and sample locations will be included in the trip report, as appropriate.

ANALYTICAL REPORT

An analytical report will be prepared for samples analyzed under this plan. Information regarding the analytical methods or procedures employed, sample results, QA/QC results, chain of custody documentation, laboratory correspondence, and raw data will be provided within this deliverable.

DATA REVIEW

A review of the data generated under this plan will be undertaken. The assessment of data acceptability or usability will be provided separately, or as part of the analytical report.

In order to establish the level of consistency achieved by the XRF unit, a correlation between confirmation sample results and XRF field screening results will be made. A regression analysis of the two data sets will be performed. In order to meet EPA QA/QC Level 2 data requirements, the regression analysis must yield correlation coefficient (r^2) of greater than 0.7. The r^2 value resulting from the regression analysis indicates how close the relationship between the two analytical methods is to being linear. A perfect linear relationship would yield an r^2 value of 1. Although the regression is not an indicator of accuracy directly, it is an indicator of how accurately you could predict laboratory results from the relationship established between the laboratory data and the XRF field screening results. As an indicator of overall accuracy, RST will calculate the average relative percent difference between the two data sets.

9.2 Data Validation

Data generated under this QA/QC Sampling Plan will be evaluated according to criteria contained in the Removal Program Data Validation Procedures that accompany OSWER Directive number 9360.4-1 and in accordance with Region II guidelines.

10.0 FIELD QUALITY CONTROL CHECKS AND FREQUENCY

In addition to the following, the Field Quality Control Checks and Frequency procedure will be conducted in accordance with Section B7 of the Region II RST QAPP. This section details the Quality Assurance/Quality Control (QA/QC) requirements for field activities performed during the sampling effort.

The following QA Protocols for XRF data are applicable to all XRF samples and include:

1. Daily instrument checks (Energy Calibration Check, Resolution Check and Zero Check).
2. Initial and continuing analysis of standards. The XRF result for medium and high standards shall be within 20% of the certified concentration of each contaminant of concern. Low, medium and high standards will be analyzed at the beginning of each screening day, at the termination of sample screening every day and following every tenth field sample analyzed.
3. Duplicate XRF analysis to be performed on at least 10% of all samples to determine the precision of sample preparation. The precision is expressed in terms of the Relative Percent Difference (RPD). A duplicate sample is a second XRF cup from the sample point.
4. Replicate XRF analysis to be performed on at least 10% of all samples to determine analytical precision. The precision is expressed in terms of the RPD for two replicate analyses, or the Relative Standard Deviation (RSD) for three replicate analyses. A replicate is a re-analysis of a single XRF cup.
5. For this sampling event, the OSC has requested that approximately 20% of the samples screened in the field using XRF be analyzed by an outside laboratory via Inductively Coupled Plasma (ICP) analysis.

Precision

Precision during XRF analysis is monitored by analyzing the NIST SRM standards at the start and the end of sample analysis and after approximately every tenth sample. Determining the precision around the site action level can be extremely important if the XRF results are to be used in an enforcement action.

For XRF results, ideally, the sample cup (if used) that was analyzed by XRF should be the same sample that is sent for confirmatory analysis. However, since the samples are being submitted for TAL Metals analysis, this will not be sufficient volume. An 8oz. jar of soil from the original homogenized aliquot

will be submitted for TAL Metals analysis. When confirming an in-situ analysis, collect a sample from a 12 inch by 12 inch area for both an XRF measurement and confirmation analysis.

QC Procedures for use of EnSys PCB Soil Test System

The Ensys PCB Soil Test Kit uses a rapid immunoassay screening method to identify PCBs in soil. A four phase method is used, and includes: extraction and preparation of the sample, sample and standard preparation, immunoassay, and analysis of results.

As part of the QA the Kit analyzes 10 soil samples per batch, plus two standards. Standards are run in duplicate with each kit, and therefore with every 10 samples that are analyzed. The standards that are run with each kit are compared using a photometer to ensure that they are within the QC limit < -0.3 .

11.0 SYSTEM AUDIT

In addition to the following, the System Audit procedure will be conducted in accordance with Section C1 of the Region II RST QAPP.

The Field QA/QC Officer will observe sampling operations and review subsequent analytical results to ensure compliance with the QA/QC requirements of the project/sampling event.

12.0 CORRECTIVE ACTION

In addition to the following, the Corrective Action procedure will be conducted in accordance with Section C1 of the Region II RST QAPP.

All provisions will be taken in the field and laboratory to ensure that any problems that may develop will be dealt with as quickly as possible to ensure the continuity of the project/sampling events. Any deviations from this sampling plan will be noted in the final report.

ATTACHMENT A
SITE LOCATION MAP



FIGURE 1
SITE LOCATION MAP
MATTEO IRON AND METAL SITE
WEST DEPTFORD, NJ

US ENVIRONMENTAL PROTECTION AGENCY
 REMOVAL SUPPORT TEAM
 CONTRACT # 68-W-00-113



Weston Solutions Inc.
FEDERAL PROGRAMS DIVISION

IN ASSOCIATION WITH SCIENTIFIC ENVIRONMENTAL ASSOCIATES, INC.
 TERRANEARPMC,
 AND INNOVATIVE TECHNOLOGICAL SOLUTIONS INC.

EDITED BY: W. HENSBERGER
 EPA OSC: N. MAGRIPLES
 SITE PROJECT MANAGER: A. LEVY
 FILE: D:\DWG\MATTEO1

ATTACHMENT B

EPA/ERT SAMPLING SOPS

SOP # 2012 - Soil Sampling

SOP # 2009 - Drum Sampling

SOP # 2011 - Chip, Wipe & Sweep Sampling

**EPA/REAC SOP# 1700- Niton XL722S Field Portable X-Ray Fluorescence Operating Procedure
(Draft)**



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- 4.0 POTENTIAL PROBLEMS
- 5.0 EQUIPMENT
- 6.0 REAGENTS
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 - 7.1 Preparation
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 - 7.2.1 Surface Soil Samples
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SUPERCEDES: SOP #2012; Revision 0.0; 11/16/94; U.S. EPA Contract 68-C4-0022.



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1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to describe the procedures for the collection of representative soil samples. Sampling depths are assumed to be those that can be reached without the use of a drill rig, direct-push, or other mechanized equipment (except for a back-hoe). Analysis of soil samples may determine whether concentrations of specific pollutants exceed established action levels, or if the concentrations of pollutants present a risk to public health, welfare, or the environment.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the actual procedures used should be documented and described in an appropriate site report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

Soil samples may be collected using a variety of methods and equipment depending on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type. Near-surface soils may be easily sampled using a spade, trowel, and scoop. Sampling at greater depths may be performed using a hand auger, continuous flight auger, a trier, a split-spoon, or, if required, a backhoe.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Chemical preservation of solids is not generally recommended. Samples should, however, be cooled and protected from sunlight to minimize any potential reaction. The amount of sample to be collected and proper sample container type are discussed in ERT/REAC SOP #2003 Rev. 0.0 08/11/94, *Sample Storage, Preservation and Handling*.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

There are two primary potential problems associated with soil sampling - cross contamination of samples and improper sample collection. Cross contamination problems can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection can involve using contaminated equipment, disturbance of the matrix resulting in compaction of the sample, or inadequate homogenization of the samples where required, resulting in variable, non-representative results.

5.0 EQUIPMENT



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Soil sampling equipment includes the following:

- Maps/plot plan
- Safety equipment, as specified in the site-specific Health and Safety Plan
- Survey equipment or global positioning system (GPS) to locate sampling points
- Tape measure
- Survey stakes or flags
- Camera and film
- Stainless steel, plastic, or other appropriate homogenization bucket, bowl or pan
- Appropriate size sample containers
- Ziplock plastic bags
- Logbook
- Labels
- Chain of Custody records and custody seals
- Field data sheets and sample labels
- Cooler(s)
- Ice
- Vermiculite
- Decontamination supplies/equipment
- Canvas or plastic sheet
- Spade or shovel
- Spatula
- Scoop
- Plastic or stainless steel spoons
- Trowel(s)
- Continuous flight (screw) auger
- Bucket auger
- Post hole auger
- Extension rods
- T-handle
- Sampling trier
- Thin wall tube sampler
- Split spoons
- Vehimeyer soil sampler outfit
 - Tubes
 - Points
 - Drive head
 - Drop hammer
 - Puller jack and grip
- Backhoe

6.0 REAGENTS



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Reagents are not used for the preservation of soil samples. Decontamination solutions are specified in ERT/REAC SOP #2006 Rev. 0.0 08/11/94, *Sampling Equipment Decontamination*, and the site specific work plan.

7.0 PROCEDURES

7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required.
2. Obtain necessary sampling and monitoring equipment.
3. Decontaminate or pre-clean equipment, and ensure that it is in working order.
4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate.
5. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety Plan.
6. Use stakes, flagging, or buoys to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All staked locations should be utility-cleared by the property owner or the On-Scene-Coordinator (OSC) prior to soil sampling; and utility clearance should always be confirmed before beginning work.

7.2 Sample Collection

7.2.1 Surface Soil Samples

Collection of samples from near-surface soil can be accomplished with tools such as spades, shovels, trowels, and scoops. Surface material is removed to the required depth and a stainless steel or plastic scoop is then used to collect the sample.

This method can be used in most soil types but is limited to sampling at or near the ground surface. Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sample team member. A flat, pointed mason trowel to cut a block of the desired soil is helpful when undisturbed profiles are required. Tools plated with chrome or other materials should not be used. Plating is particularly common with garden implements such as potting trowels.

The following procedure is used to collect surface soil samples:



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1. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.
2. Using a pre-cleaned, stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade.
3. If volatile organic analysis is to be performed, transfer the sample directly into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval or location into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

7.2.2 Sampling at Depth with Augers and Thin Wall Tube Samplers

This system consists of an auger, or a thin-wall tube sampler, a series of extensions, and a "T" handle (Figure 1, Appendix A). The auger is used to bore a hole to a desired sampling depth, and is then withdrawn. The sample may be collected directly from the auger. If a core sample is to be collected, the auger tip is then replaced with a thin wall tube sampler. The system is then lowered down the borehole, and driven into the soil to the completion depth. The system is withdrawn and the core is collected from the thin wall tube sampler.

Several types of augers are available; these include: bucket type, continuous flight (screw), and post-hole augers. Bucket type augers are better for direct sample recovery because they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the flights. The continuous flight augers are satisfactory when a composite of the complete soil column is desired. Post-hole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy soil and cannot be used below a depth of approximately three feet.

The following procedure is used for collecting soil samples with the auger:

1. Attach the auger bit to a drill rod extension, and attach the "T" handle to the drill rod.



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2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first three to six inches of surface soil for an area approximately six inches in radius around the drilling location.
3. Begin augering, periodically removing and depositing accumulated soils onto a plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole, and avoids possible contamination of the surrounding area.
4. After reaching the desired depth, slowly and carefully remove the auger from the hole. When sampling directly from the auger, collect the sample after the auger is removed from the hole and proceed to Step 10.
5. Remove auger tip from the extension rods and replace with a pre-cleaned thin wall tube sampler. Install the proper cutting tip.
6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the soil. Do not scrape the borehole sides. Avoid hammering the rods as the vibrations may cause the boring walls to collapse.
7. Remove the tube sampler, and unscrew the drill rods.
8. Remove the cutting tip and the core from the device.
9. Discard the top of the core (approximately 1 inch), as this possibly represents material collected before penetration of the layer of concern. Place the remaining core into the appropriate labeled sample container. Sample homogenization is not required.
10. If volatile organic analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly.

When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.



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11. If another sample is to be collected in the same hole, but at a greater depth, reattach the auger bit to the drill and assembly, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.
12. Abandon the hole according to applicable state regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

7.2.3 Sampling with a Trier

The system consists of a trier, and a "T" handle. The auger is driven into the soil to be sampled and used to extract a core sample from the appropriate depth.

The following procedure is used to collect soil samples with a sampling trier:

1. Insert the trier (Figure 2, Appendix A) into the material to be sampled at a 0° to 45° angle from horizontal. This orientation minimizes the spillage of sample.
2. Rotate the trier once or twice to cut a core of material.
3. Slowly withdraw the trier, making sure that the slot is facing upward.
4. If volatile organic analyses are required, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

7.2.4 Sampling at Depth with a Split Spoon (Barrel) Sampler

Split spoon sampling is generally used to collect undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted with a split spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augured hole and the core extracted.

When split spoon sampling is performed to gain geologic information, all work should



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be performed in accordance with ASTM D1586-98, "Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils".

The following procedures are used for collecting soil samples with a split spoon:

1. Assemble the sampler by aligning both sides of barrel and then screwing the drive shoe on the bottom and the head piece on top.
2. Place the sampler in a perpendicular position on the sample material.
3. Using a well ring, drive the tube. Do not drive past the bottom of the head piece or compression of the sample will result.
4. Record in the site logbook or on field data sheets the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth.
5. Withdraw the sampler, and open by unscrewing the bit and head and splitting the barrel. The amount of recovery and soil type should be recorded on the boring log. If a split sample is desired, a cleaned, stainless steel knife should be used to divide the tube contents in half, longitudinally. This sampler is typically available in 2 and 3 1/2 inch diameters. A larger barrel may be necessary to obtain the required sample volume.
6. Without disturbing the core, transfer it to appropriate labeled sample container(s) and seal tightly.

7.2.5 Test Pit/Trench Excavation

A backhoe can be used to remove sections of soil, when detailed examination of soil characteristics are required. This is probably the most expensive sampling method because of the relatively high cost of backhoe operation.

The following procedures are used for collecting soil samples from test pits or trenches:

1. Prior to any excavation with a backhoe, it is important to ensure that all sampling locations are clear of overhead and buried utilities.
2. Review the site specific Health & Safety plan and ensure that all safety precautions including appropriate monitoring equipment are installed as required.



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3. Using the backhoe, excavate a trench approximately three feet wide and approximately one foot deep below the cleared sampling location. Place excavated soils on plastic sheets. Trenches greater than five feet deep must be sloped or protected by a shoring system, as required by OSHA regulations.
4. A shovel is used to remove a one to two inch layer of soil from the vertical face of the pit where sampling is to be done.
5. Samples are taken using a trowel, scoop, or coring device at the desired intervals. Be sure to scrape the vertical face at the point of sampling to remove any soil that may have fallen from above, and to expose fresh soil for sampling. In many instances, samples can be collected directly from the backhoe bucket.
6. If volatile organic analyses are required, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.
7. Abandon the pit or excavation according to applicable state regulations. Generally, shallow excavations can simply be backfilled with the removed soil material.

8.0 CALCULATIONS

This section is not applicable to this SOP.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

There are no specific quality assurance (QA) activities which apply to the implementation of these procedures. However, the following QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.
2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration



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activities must occur prior to sampling/operation, and they must be documented.

10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA and corporate health and safety procedures, in addition to the procedures specified in the site specific Health & Safety Plan..

12.0 REFERENCES

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Barth, D.S. and B.J. Mason. 1984. Soil Sampling Quality Assurance User's Guide. EPA-600/4-84-043.

U.S. Environmental Protection Agency. 1984 Characterization of Hazardous Waste Sites - A Methods Manual: Volume II. Available Sampling Methods, Second Edition. EPA-600/4-84-076.

de Vera, E.R., B.P. Simmons, R.D. Stephen, and D.L. Storm. 1980. Samplers and Sampling Procedures for Hazardous Waste Streams. EPA-600/2-80-018.

ASTM D 1586-98, ASTM Committee on Standards, Philadelphia, PA.



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APPENDIX A
Figures
SOP #2012
February 2000



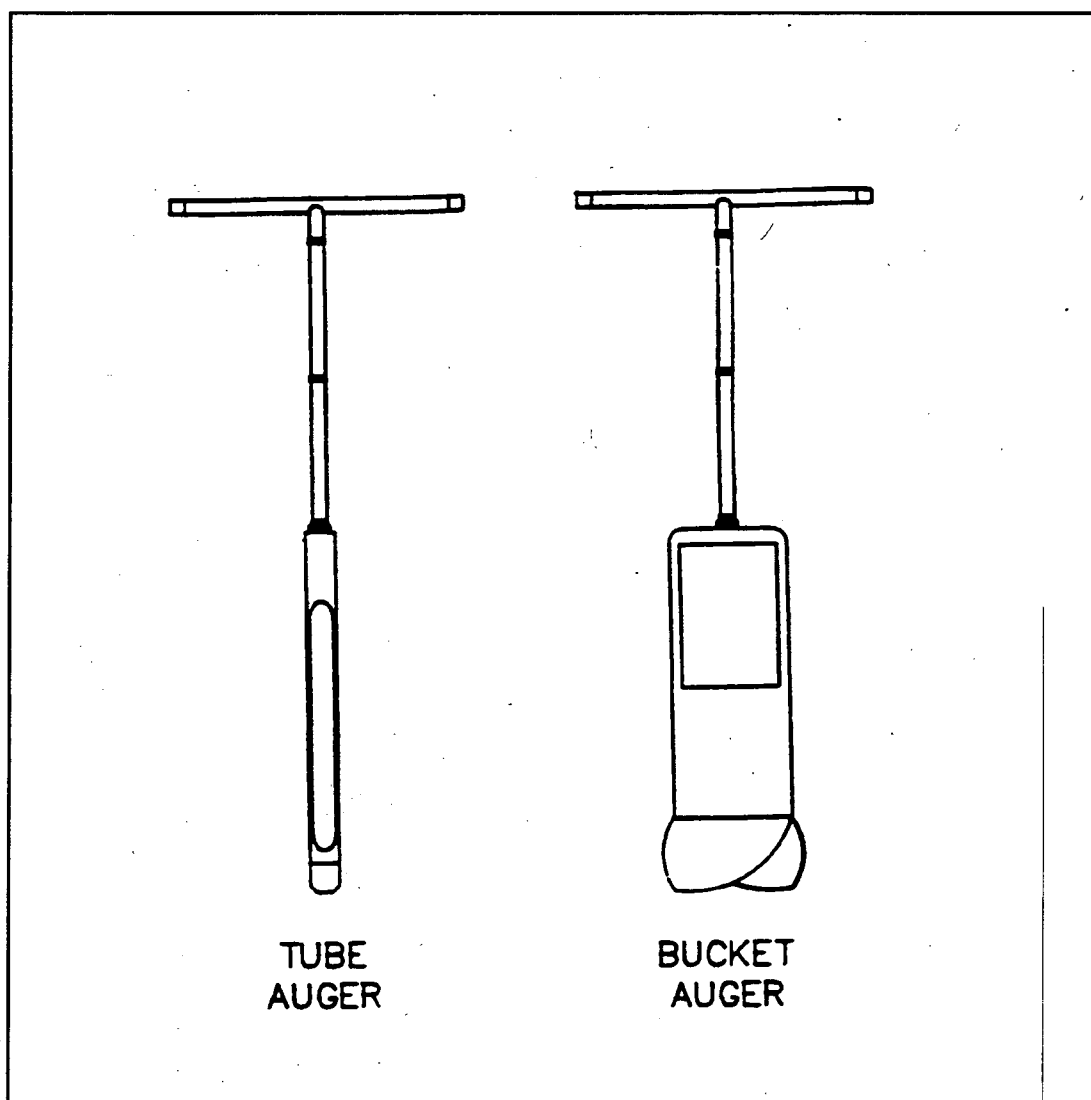
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FIGURE 1. Sampling Augers





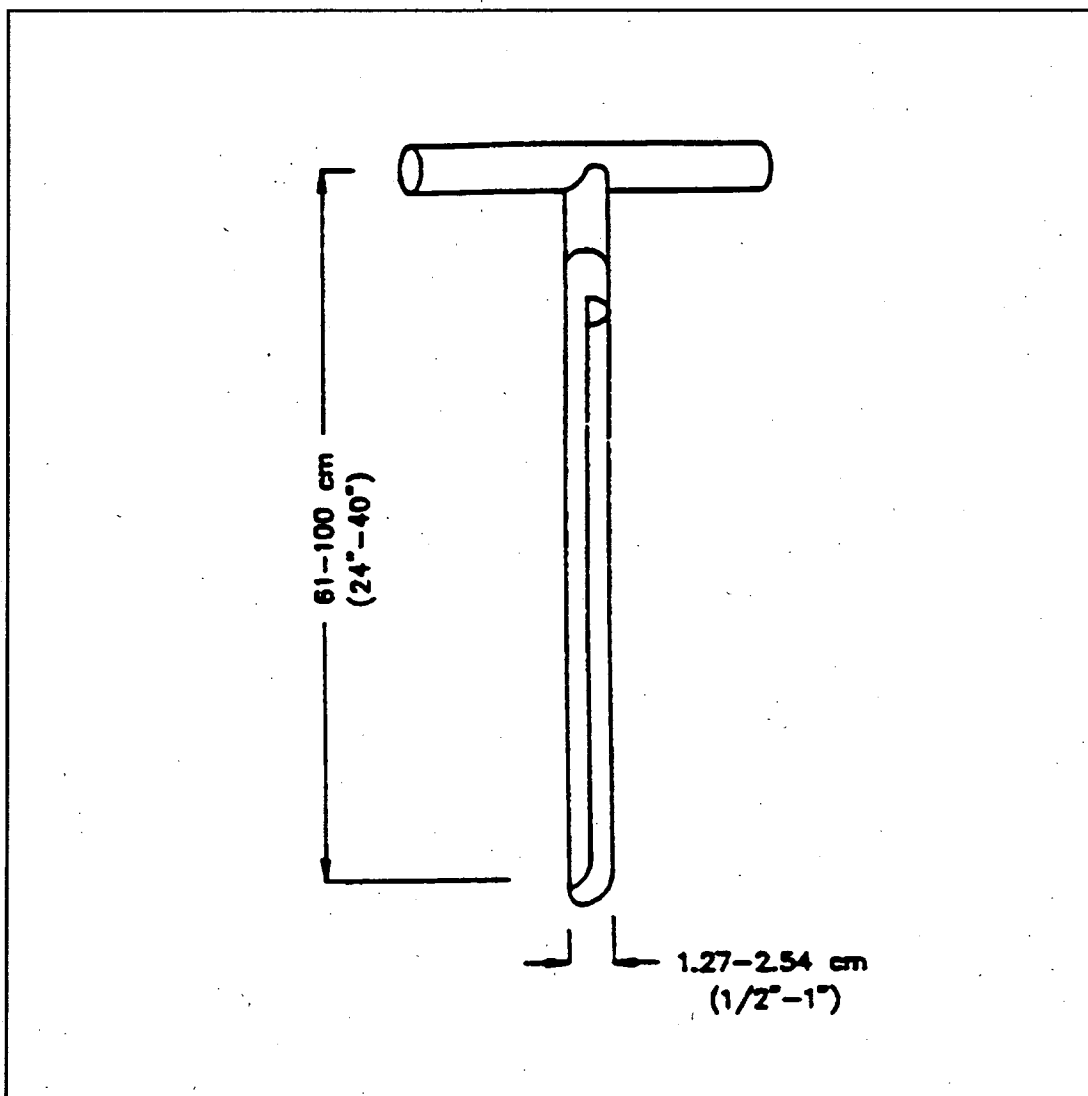
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SOIL SAMPLING

FIGURE 2. Sampling Trier





DRUM SAMPLING

SOP#: 2009
DATE: 11/16/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to provide technical guidance on implementing safe and cost-effective response actions at hazardous waste sites containing drums with unknown contents. Container contents are sampled and characterized for disposal, bulking, recycling, segregation, and classification purposes.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (U.S. EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

Prior to sampling, drums must be excavated, (if necessary), inspected, staged, and opened. Drum excavation must be performed by qualified personnel. Inspection involves the observation and recording of visual qualities of each drum and any characteristics pertinent to the classification of the drum's contents. Staging involves the physical grouping of drums according to classifications established during the physical inspection. Opening of closed drums can be performed manually or remotely. Remote drum opening is recommended for worker safety. The most widely used method of sampling a drum involves the use of a glass thief. This method is quick, simple, relatively inexpensive, and requires no decontamination. The contents of a drum can be further characterized by performing various field tests.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Samples collected from drums are considered waste samples and as such, adding preservatives is not required due to the potential reaction of the sample with the preservative. Samples should, however, be cooled to 4°C and protected from sunlight in order to minimize any potential reaction due to the light sensitivity of the sample.

Sample bottles for collection of waste liquids, sludges, or solids are typically wide mouth amber jars with Teflon-lined screw caps. Actual volume required for analysis should be determined in conjunction with the laboratory performing the analysis.

Waste sample handling procedures should be as follows:

1. Label the sample container with the appropriate sample label and complete the appropriate field data sheet(s). Place sample container into two resealable plastic bags.
2. Place each bagged sample container into a shipping container which has been lined with plastic. Pack the container with enough non-combustible, absorbent, cushioning material to minimize the possibility of containers breaking, and to absorb any material which may leak.

Note: Depending on the nature and quantity of the material to be shipped, different packaging may be required. The transportation company or a shipping/receiving expert should be consulted prior to packing the samples.

3. Complete a chain of custody record for each shipping container, place into a resealable

plastic bag, and affix to the inside lid of the shipping container.

4. Secure and custody seal the lid of the shipping container. Label the shipping container appropriately and arrange for the appropriate transportation mode consistent with the type of hazardous waste involved.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

If buried drums are suspected, geophysical investigation techniques such as magnetometry or ground penetrating radar may be employed in an attempt to determine the location and depth of drums. During excavation, the soil must be removed with great caution to minimize the potential for drum rupture.

Until the contents are characterized, sampling personnel should assume that unlabelled drums contain hazardous materials. Labelled drums are frequently mislabelled, especially drums that are reused. Because a drum's label may not accurately describe its contents, extreme caution must be exercised when working with or around drums.

If a drum which contains a liquid cannot be moved without rupture, its contents may be immediately transferred to a sound drum using an appropriate method of transfer based on the type of waste. In any case, preparations should be made to contain the spill (i.e., spill pads, dike, etc.) should one occur.

If a drum is leaking, open, or deteriorated, then it must be placed immediately in overpack containers.

The practice of tapping drums to determine their contents is neither safe nor effective and should not be used if the drums are visually overpressurized or if shock-sensitive materials are suspected. A laser thermometer may be effective in order to determine the level of the drum contents via surface temperature differences.

Drums that have been overpressurized to the extent that the head is swollen several inches above the level of the chime should not be moved. A number of devices have been developed for venting critically swollen drums. One method that has proven to be effective is a tube and spear device. A light aluminum

tube (3 meters long) is positioned at the vapor space of the drum. A rigid, hooking device attached to the tube, goes over the chime and holds the tube securely in place. The spear is inserted in the tube and positioned against the drum wall. A sharp blow on the end of the spear drives the sharpened tip through the drum and the gas vents along the grooves. Venting should be done from behind a wall or barricade. Once the pressure has been relieved, the bung can be removed and the drum sampled.

Because there is potential for accidents to occur during handling, particularly initial handling, drums should only be handled if necessary. All personnel should be warned of the hazards prior to handling drums. Overpack drums and an adequate volume of absorbent material should be kept near areas where minor spills may occur. Where major spills may occur, a containment berm adequate to contain the entire volume of liquid in the drums should be constructed before any handling takes place. If drum contents spill, personnel trained in spill response should be used to isolate and contain the spill.

5.0 EQUIPMENT/APPARATUS

The following are standard materials and equipment required for sampling:

- Personal protection equipment
- Wide-mouth amber glass jars with Teflon cap liner, approximately 500 mL volume
- Other appropriate sample jars
- Uniquely numbered sample identification labels with corresponding data sheets
- Drum/Tank Sampling Data Sheets and Field Test Data Sheets for Drum/Tank Sampling
- Chain of Custody records
- Decontamination materials
- Glass thieving tubes or COLIWASA
- Coring device
- Stainless steel spatula or spoons
- Laser thermometer
- Drum overpacks
- Absorbent material for spills
- Drum opening devices

Bung Wrench

A common method for opening drums manually is using a universal bung wrench. These wrenches have fittings made to

remove nearly all commonly encountered bungs. They are usually constructed of a non-sparking metal alloy (i.e., brass, bronze/manganese, aluminum, etc.) formulated to reduce the likelihood of sparks. The use of a "NON-SPARKING" wrench does not completely eliminate the possibility of a spark being produced.

Drum Deheader

One means by which a drum can be opened manually when a bung is not removable with a bung wrench is by using a drum deheader. This tool is constructed of forged steel with an alloy steel blade and is designed to cut the lid of a drum off or part way off by means of a scissors-like cutting action. A limitation of this device is that it can be attached only to closed head drums. Drums with removable heads must be opened by other means.

Hand Pick, Pickaxe, and Hand Spike

These tools are usually constructed of brass or a non-sparking alloy with a sharpened point that can penetrate the drum lid or head when the tool is swung. The hand picks or pickaxes that are most commonly used are commercially available; whereas, the spikes are generally uniquely fabricated four foot long poles with a pointed end.

Backhoe Spike

Another means used to open drums remotely for sampling is a metal spike attached or welded to a backhoe bucket. This method is very efficient and is often used in large-scale operations.

Hydraulic Drum Opener

Recently, remotely operated hydraulic devices have been fabricated to open drums. This device uses hydraulic pressure to force a non-sparking spike through the wall of a drum. It consists of a manually operated pump which pressurizes fluid through a length of hydraulic line.

Pneumatic Devices

A pneumatic bung remover consists of a compressed air supply that is controlled by a two-stage regulator. A high pressure air line of desired length delivers compressed air to a pneumatic drill, which is adapted to turn bung fitting selected to fit the bung to be removed. An adjustable bracketing system has been designed to position and align the pneumatic drill over the bung. This bracketing system must be attached to the drum before the drill can be operated. Once the bung has been loosened, the bracketing system must be removed before the drum can be sampled. This remote bung opener does not permit the slow venting of the container, and therefore appropriate precautions must be taken. It also requires the container to be upright and relatively level. Bungs that are rusted shut cannot be removed with this device.

6.0 REAGENTS

Reagents are not typically required for preserving drum samples. However, reagents will be utilized for decontamination of sampling equipment.

7.0 PROCEDURES

7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.
2. Obtain necessary sampling and monitoring equipment.
3. Decontaminate or preclean equipment, and ensure that it is in working order.
4. Prepare scheduling and coordinate with staff, clients, and regulatory agency, if appropriate.
5. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety Plan.

6. Use stakes, flagging, or buoys to identify and mark all sampling locations. If required the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

7.2 Drum Excavation

If it is presumed that buried drums are on-site and prior to beginning excavation activities, geophysical investigation techniques should be utilized to approximate the location and depth of the drums. In addition, it is important to ensure that all locations where excavation will occur are clear of utility lines, pipes and poles (subsurface as well as above surface).

Excavating, removing, and handling drums are generally accomplished with conventional heavy construction equipment. These activities should be performed by an equipment operator who has experience in drum excavation. During excavation activities, drums must be approached in a manner that will avoid digging directly into them.

The soil around the drum should be excavated with non-sparking hand tools or other appropriate means and as the drums are exposed, a visual inspection should be made to determine the condition of the drums. Ambient air monitoring should be done to determine the presence of unsafe levels of volatile organics, explosives, or radioactive materials. Based on this preliminary visual inspection, the appropriate mode of drum excavation and handling may be determined.

Drum identification and inventory should begin before excavation. Information such as location, date of removal, drum identification number, overpack status, and any other identification marks should be recorded on the Drum/Tank Sampling Data Sheet (Attachment 1, Appendix A).

7.3 Drum Inspection

Appropriate procedures for handling drums depend on the contents. Thus, prior to any handling, drums should be visually inspected to gain as much information as possible about their contents. The drums should be inspected for the following:

1. Drum condition, corrosion, rust, punctures, bungs, and leaking contents.

2. Symbols, words, or other markings on the drum indicating hazards (i.e., explosive, radioactive, toxic, flammable), or further identifying the drums.
3. Signs that the drum is under pressure.
4. Shock sensitivity.

Monitoring should be conducted around the drums using instruments such as radiation meters, organic vapor analyzers (OVA) and combustible gas indicators (CGI).

Survey results can be used to classify the drums into categories, for instance:

- Radioactive
- Leaking/deteriorating
- Bulging
- Lab packs
- Explosive/shock sensitive
- Empty

All personnel should assume that unmarked drums contain hazardous materials until their contents have been categorized. Once a drum has been visually inspected and any immediate hazard has been eliminated by overpacking or transferring the drum's contents, the drum is affixed with a numbered tag and transferred to a staging area. Color-coded tags, labels or bands should be used to identify the drum's category based on visual inspection. A description of each drum, its condition, any unusual markings, the location where it was buried or stored, and field monitoring information are recorded on a Drum/Tank Sampling Data Sheet (Attachment 1, Appendix A). This data sheet becomes the principal record keeping tool for tracking the drum on-site.

7.4 Drum Staging

Prior to sampling, the drums should be staged to allow easy access. Ideally, the staging area should be located just far enough from the drum opening area to prevent a chain reaction if one drum should explode or catch fire when opened.

During staging, the drums should be physically separated into the following categories: those containing liquids, those containing solids, those containing lab packs, and those which are empty.

This is done because the strategy for sampling and handling drums/containers in each of these categories will be different. This may be achieved by visual inspection of the drum and its labels, codes, etc. Solids and sludges are typically disposed of in open top drums. Closed head drums with a bung opening generally contain liquid.

Where there is good reason to suspect that drums contain radioactive, explosive, or shock-sensitive materials, these drums should be staged in a separate, isolated area. Placement of explosives and shock-sensitive materials in diked and fenced areas will minimize the hazard and the adverse effects of any premature detonation of explosives.

Where space allows, the drum opening area should be physically separated from the drum removal and drum staging operations. Drums are moved from the staging area to the drum opening area one at a time using forklift trucks equipped with drum grabbers or a barrel grapppler. In a large-scale drum handling operation, drums may be conveyed to the drum opening area using a roller conveyor. Drums may be restaged as necessary after opening and sampling.

7.5 Drum Opening

There are three basic techniques available for opening drums at hazardous waste sites:

- Manual opening with non-sparking bung wrenches
- Drum deheading
- Remote drum puncturing or bung removal

The choice of drum opening techniques and accessories depends on the number of drums to be opened, their waste contents, and physical condition. Remote drum opening equipment should always be considered in order to protect worker safety. Under OSHA 1910.120, manual drum opening with bung wrenches or deheaders should be performed ONLY with structurally sound drums and waste contents that are known to be non-shock sensitive, non-reactive, non-explosive, and non-flammable.

7.5.1 Manual Drum Opening with a Bung Wrench

Manual drum opening with bung wrenches (Figure 1, Appendix B) should not be performed unless the

drums are structurally sound (no evidence of bulging or deformation) and their contents are known to be non-shock sensitive, non-reactive, non-explosive or non-flammable. If opening the drum with bung wrenches is deemed safe, then certain procedures should be implemented to minimize the hazard:

- Field personnel should be fully outfitted with protective gear.
- Drums should be positioned upright with the bung up, or, for drums with bungs on the side, laid on their sides with the bung plugs up.
- The wrenching motion should be a slow, steady pull across the drum. If the length of the bung wrench handle provides inadequate leverage for unscrewing the plug, a "cheater bar" can be attached to the handle to improve leverage.

7.5.2 Manual Drum Opening with a Drum Deheader

Drums are opened with a drum deheader (Figure 2, Appendix B) by first positioning the cutting edge just inside the top chime and then tightening the adjustment screw so that the deheader is held against the side of the drum. Moving the handle of the deheader up and down while sliding the deheader along the chime will enable the entire top to be rapidly cut off if so desired. If the top chime of a drum has been damaged or badly dented it may not be possible to cut the entire top off. Since there is always the possibility that a drum may be under pressure, the initial cut should be made very slowly to allow for the gradual release of any built-up pressure. A safer technique would be to employ a remote method prior to using the deheader.

Self-propelled drum openers which are either electrically or pneumatically driven are available and can be used for quicker and more efficient deheading.

The drum deheader should be decontaminated, as necessary, after each drum is opened to avoid cross contamination and/or adverse chemical reactions from incompatible materials.

7.5.3 Manual Drum Opening with a Hand Pick, Pickaxe, or Spike

When a drum must be opened and neither a bung

wrench nor a drum deheader is suitable, then it can be opened for sampling by using a hand pick, pickaxe, or spike (Figure 3, Appendix B). Often the drum lid or head must be hit with a great deal of force in order to penetrate it. Because of this, the potential for splash or spraying is greater than with other opening methods and therefore, this method of drum opening is not recommended, particularly when opening drums containing liquids. Some spikes used have been modified by the addition of a circular splash plate near the penetrating end. This plate acts as a shield and reduces the amount of splash in the direction of the person using the spike. Even with this shield, good splash gear is essential.

Since drums, some of which may be under pressure, cannot be opened slowly with these tools, spray from drums is common and appropriate safety measures must be taken. The pick or spike should be decontaminated after each drum is opened to avoid cross contamination and/or adverse chemical reaction from incompatible materials.

7.5.4 Remote Drum Opening with a Backhoe Spike

Remotely operated drum opening tools are the safest available means of drum opening. Remote drum opening is slow, but provides a high degree of safety compared to manual methods of opening.

In the opening area, drums should be placed in rows with adequate aisle space to allow ease in backhoe maneuvering. Once staged, the drums can be quickly opened by punching a hole in the drum head or lid with the spike.

The spike (Figure 4, Appendix B) should be decontaminated after each drum is opened to prevent cross contamination and/or adverse reaction from incompatible material. Even though some splash or spray may occur when this method is used, the operator of the backhoe can be protected by mounting a large shatter-resistant shield in front of the operator's cage. This combined with the normal personal protection gear should be sufficient to protect the operator. Additional respiratory protection can be afforded by providing the operator with an on-board airline system.

7.5.5 Remote Drum Opening with Hydraulic Devices

A piercing device with a non-sparking, metal point is attached to the end of a hydraulic line and is pushed into the drum by the hydraulic pressure (Figure 5, Appendix B). The piercing device can be attached so that a hole for sampling can be made in either the side or the head of the drum. Some of the metal piercers are hollow or tube-like so that they can be left in place if desired and serve as a permanent tap or sampling port. The piercer is designed to establish a tight seal after penetrating the container.

7.5.6 Remote Drum Opening with Pneumatic Devices

Pneumatically-operated devices utilizing compressed air have been designed to remove drum bungs remotely (Figure 6, Appendix B). Prior to opening the drum, a bung fitting must be selected to fit the bung to be removed. The adjustable bracketing system is then attached to the drum and the pneumatic drill is aligned over the bung. This must be done before the drill can be operated. The operator then moves away from the drum to operate the equipment. Once the bung has been loosened, the bracketing system must be removed before the drum can be sampled. This remote bung opener does not permit the slow venting of the container, and therefore appropriate precautions must be taken. It also requires the container to be upright and relatively level. Bungs that are rusted shut cannot be removed with this device.

7.6 Drum Sampling

After the drum has been opened, preliminary monitoring of headspace gases should be performed first with an explosimeter/oxygen meter. Afterwards, an OVA or other instruments should be used. If possible, these instruments should be intrinsically safe. In most cases it is impossible to observe the contents of these sealed or partially sealed drums. Since some layering or stratification is likely in any solution left undisturbed, a sample that represents the entire depth of the drum must be taken.

When sampling a previously sealed drum, a check should be made for the presence of a bottom sludge. This is easily accomplished by measuring the depth to apparent bottom then comparing it to the known interior depth.

7.6.1 Glass Thief Sampler

The most widely used implement for sampling drum liquids is a glass tube commonly referred to as a glass thief (Figure 7, Appendix B). This tool is cost effective, quick, and disposable. Glass thieves are typically 6mm to 16mm I.D. and 48 inches long.

Procedures for Use:

1. Remove the cover from the sample container.
2. Insert glass tubing almost to the bottom of the drum or until a solid layer is encountered. About one foot of tubing should extend above the drum.
3. Allow the waste in the drum to reach its natural level in the tube.
4. Cap the top of the sampling tube with a tapered stopper or thumb, ensuring liquid does not come into contact with stopper.
5. Carefully remove the capped tube from the drum and insert the uncapped end into the appropriate sample container.
6. Release stopper and allow the glass thief to drain until the container is approximately two-thirds full.
7. Remove tube from the sample container, break it into pieces and place the pieces in the drum.
8. Cap the sample container tightly and label it. Place the sample container into a carrier.
9. Replace the bung or place plastic over the drum.
10. Log all samples in the site logbook and on Drum/Tank Sampling Data Sheets.
11. Perform hazard categorization analyses if included in the project scope.
12. Transport the sample to the decontamination zone and package it for transport to the analytical laboratory, as necessary. Complete chain of custody records.

In many instances a drum containing waste material will have a sludge layer on the bottom. Slow insertion of the sample tube into this layer; then a gradual withdrawal will allow the sludge to act as a bottom plug to maintain the fluid in the tube. The plug can be gently removed and placed into the sample container by the use of a stainless steel lab spoon.

It should be noted that in some instances disposal of the tube by breaking it into the drum may interfere with eventual plans for the removal of its contents. The use of this technique should be cleared with the project officer or other glass thief disposal techniques should be evaluated.

7.6.2 COLIWASA Sampler

The Composite Liquid Waste Sampler (COLIWASA) and modifications thereof are equipment that collect a sample from the full depth of a drum and maintain it in the transfer tube until delivery to the sample bottle. The COLIWASA (Figure 8, Appendix B) is a much cited sampler designed to permit representative sampling of multiphase wastes from drums and other containerized wastes. One configuration consists of a 152 cm by 4 cm I.D. section of tubing with a neoprene stopper at one end attached by a rod running the length of the tube to a locking mechanism at the other end.

Manipulation of the locking mechanism opens and closes the sampler by raising and lowering the neoprene stopper. One model of the COLIWASA is shown in Appendix B; however, the design can be modified and/or adapted somewhat to meet the needs of the sampler.

The major drawbacks associated with using a COLIWASA concern decontamination and costs. The sampler is difficult to decontaminate in the field and its high cost in relation to alternative procedures (glass tubes) make it an impractical throwaway item. It still has applications, however, especially in instances where a true representation of a multiphase waste is absolutely necessary.

Procedures for Use

1. Put the sampler in the open position by placing the stopper rod handle in the T-position and pushing the rod down until the handle sits against the sampler's locking block.

2. Slowly lower the sampler into the liquid waste. Lower the sampler at a rate that permits the levels of the liquid inside and outside the sampler tube to be about the same. If the level of the liquid in the sample tube is lower than that outside the sampler, the sampling rate is too fast and will result in a non-representative sample.
3. When the sampler stopper hits the bottom of the waste container, push the sampler tube downward against the stopper to close the sampler. Lock the sampler in the closed position by turning the T-handle until it is upright and one end rests tightly on the locking block.
4. Slowly withdraw the sample from the waste container with one hand while wiping the sampler tube with a disposable cloth or rag with the other hand.
5. Carefully discharge the sample into the appropriate sample container by slowly pulling the lower end of the T-handle away from the locking block while the lower end of the sampler is positioned in a sample container.
6. Cap the sample container tightly and label it. Place the sample container in a carrier.
7. Replace the bung or place plastic over the drum.
8. Log all samples in the site logbook and on Drum/Tank Sampling Data Sheets.
9. Perform hazard categorization analyses if included in the project scope.
10. Transport the sample to the decontamination zone and package for transport to the analytical laboratory, as necessary. Complete the Chain of Custody records.

7.6.3 Coring Device

A coring device may be used to sample drum solids. Samples should be taken from different areas within the drum. This sampler consists of a series of extensions, a T-handle, and the coring device.

Procedures for use:

1. Assemble the sampling equipment.
2. Remove the cover from the sample container.
3. Insert the sampling device to the bottom of the drum. The extensions and the "T" handle should extend above the drum.
4. Rotate the sampling device to cut a core of material.
5. Slowly withdraw the sampling device so that as much sample material as possible is retained within it.
6. Transfer the sample to the appropriate sample container, and label it. A stainless steel spoon or scoop may be used as necessary.
7. Cap the sample container tightly and place it in a carrier.
8. Replace the bung or place plastic over the drum.
9. Log all samples in the site log book and on Drum/Tank Sampling Data Sheets.
10. Perform hazard categorization analyses if included in the project scope.
11. Transport the sample to the decontamination zone and package it for transport to the analytical laboratory, as necessary. Complete chain of custody records.

7.7 Hazard Categorization

The goal of characterizing or categorizing the contents of drums is to obtain a quick, preliminary assessment of the types and levels of pollutants contained in the drums. These activities generally involve rapid, non-rigorous methods of analysis. The data obtained from these methods can be used to make decisions regarding drum staging or restaging, bulking or compositing of the drum contents.

As a first step in obtaining these data, standard tests should be used to classify the drum contents into general categories such as auto-reactives, water reactives, inorganic acids, organic acids, heavy metals, pesticides, cyanides, inorganic oxidizers, and organic oxidizers. In some cases, further analyses should be conducted to more precisely identify the drum contents.

There are several methods available to perform these tests:

- the HazCat[®] chemical identification system
- the Chlor-N-Oil Test Kit
- Spill-fyter Chemical Classifier Strips
- Setaflash (for ignitability)

These methods must be performed according to the manufacturers' instructions and the results must be documented on the Field Test Data Sheet for Drum/Tank Sampling (Attachment 2, Appendix A).

Other tests which may be performed include:

- Water Reactivity
- Specific Gravity Test (compared to water)
- Water Solubility Test
- pH of Aqueous Solution

The tests must be performed in accordance with the instructions on the Field Test Data Sheet for Drum/Tank Sampling and results of the tests must be documented on these data sheets.

The specific methods that will be used for hazard categorization must be documented in the Quality Assurance Work Plan.

8.0 CALCULATIONS

This section is not applicable to this SOP.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

The following general quality assurance procedures apply:

1. All data must be documented on Chain of Custody records, Drum/Tank Sampling Data Sheets, Field Test Data Sheet for Drum/Tank Sampling, or within site logbooks.
2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.

10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety procedures.

More specifically, the opening of closed containers is one of the most hazardous site activities. Maximum efforts should be made to ensure the safety of the sampling team. Proper protective equipment and a general awareness of the possible dangers will minimize the risk inherent to sampling operations. Employing proper drum opening techniques and equipment will also safeguard personnel. The use of remote sampling equipment whenever feasible is highly recommended.

12.0 REFERENCES

Guidance Document for Cleanup of Surface Tank and Drum Sites, OSWER Directive 9380.0-3.

Drum Handling Practices at Hazardous Waste Sites, EPA-600/2-86-013.

APPENDIX A

Attachments

ATTACHMENT 1. Drum/Tank Sampling Data Sheet

Samplers: _____ Date: _____

Site Name: _____ Work Order Number: 3347-040-001- _____

Container Number/Sample Number: _____ REAC Task Leader: _____

SITE INFORMATION:

1. Terrain, drainage description: _____

2. Weather conditions (from observation): _____

MET station on site: No Yes

CONTAINER INFORMATION:

1. Container type: Drum Tank Other: _____

2. Container dimensions: Shape: _____

Approximate size: _____

3. Label present: No

Yes: _____

Other Markings: _____

4. Spill or leak present: No Yes Dimensions: _____

5. Container location: (Circle one) N/A See Map Other: _____

APPENDIX A (Cont'd)

Attachments

ATTACHMENT 1. Drum/Tank Sampling Data Sheet (cont'd)

SAMPLE INFORMATION:

1. Description: _____ liquid _____ solid (_____ powder or _____ crystals) _____ sludge

2. Color: _____ Vapors: _____

Other: _____

3. Local effects present: (damage - environmental, material) _____

FIELD MONITORING:

1. PID: _____ Background (clean zone)

_____ Probe used/Model used

_____ Reading from container opening

2. FID: _____ Background (clean zone)

_____ Reading from container opening

3. Radiation Meter:

_____ Model used

_____ Background (clean zone)

_____ Reading from container opening

4. Explosimeter/Oxygen Meter:

_____ Oxygen level from container opening

_____ LEL level from container opening

APPENDIX A (Cont'd)

Attachments

ATTACHMENT 2. Field Test Data Sheet for Drum/Tank Sampling

Samplers: _____

Date: _____

Site Name: _____

Work Order Number: 3347-040-001-_____

Container Number/Sample Number: _____

REAC Task Leader: _____

SAMPLE MONITORING INFORMATION:

1. PID: _____ Background (clean zone)

_____ Probe used/Model used

_____ Reading from sample

2. FID: _____ Background (clean zone)

_____ Reading from sample

3. Radiation Meter: _____ Model used

_____ Background (clean zone)

_____ Reading from sample

4. Explosimeter/Oxygen Meter: _____ Oxygen level (sample)

_____ LEL level (sample)

SAMPLE DESCRIPTION:

_____ Liquid _____ Solid _____ Sludge _____ Color _____ Vapors

WATER REACTIVITY:

1. Add small amount of sample to water: _____ bubbles _____ color change to _____

_____ vapor formation _____ heat _____ No Change

SPECIFIC GRAVITY TEST (compared to water):

1. Add small amount of sample to water: _____ sinks _____ floats

2. If liquid sample sinks, screen for chlorinated compounds. If liquid sample floats and appears to be oily, screen for PCBs (Chlor-N-Oil kit).

APPENDIX A (Cont'd)

Attachments

ATTACHMENT 2. Field Test Data Sheet for Drum/Tank Sampling (cont'd)

CHLOR N OIL TEST KIT INFORMATION:

1. Test kit used for this sample: Yes No
2. Results: _____ PCB not present _____ PCB present, less than 50 ppm
 _____ PCB present, greater than 50 ppm _____ 100% PCB present

WATER SOLUBILITY TEST:

1. Add approximately one part sample to five parts water. You may need to stir and heat gently. **[DO NOT HEAT IF WATER REACTIVE!]** Results: _____ total _____ partial _____ no solubility

pH OF AQUEOUS SOLUTION:

1. Using 0-14 pH paper, check pH of water/sample solution: _____.

SPILL-FYTER CHEMICAL CLASSIFIER STRIPS:

- | 1. Acid/Base Risk: (Circle one) | <u>Color Change</u> |
|---------------------------------|---------------------|
| Strong acid (0) | RED |
| Moderately acidic (1-3) | ORANGE |
| Weak acid (5) | YELLOW |
| Neutral (7) | GREEN |
| Moderately basic (9-11) | Dark GREEN |
| Strong Base (13-14) | Dark BLUE |
-
- | | |
|--------------------------------|---|
| 2. Oxidizer Risk: (Circle one) | |
| Not Present | WHITE |
| Present | BLUE, RED, OR ANY DIVERGENCE FROM WHITE |
-
- | | |
|--------------------------------|--------|
| 3. Fluoride Risk: (Circle one) | |
| Not Present | PINK |
| Present | YELLOW |

APPENDIX A (Cont'd)

Attachments

ATTACHMENT 2. Field Test Data Sheet for Drum/Tank Sampling (cont'd)

4. Petroleum Product, Organic Solvent Risk: (Circle one)

Not Present

LIGHT BLUE

Present

DARK BLUE

5. Iodine, Bromine, Chlorine Risk: (Circle one)

Not Present

PEACH

Present

WHITE OR YELLOW

SETAFLASH IGNITABILITY TEST:

140°F	Ignitable: _____	Non-Ignitable _____
160°F	Ignitable: _____	Non-Ignitable _____
_____	Ignitable: _____	Non-Ignitable _____
_____	Ignitable: _____	Non-Ignitable _____
_____	Ignitable: _____	Non-Ignitable _____
_____	Ignitable: _____	Non-Ignitable _____

Comments:

HAZCAT KIT TESTS:

1. Test: _____ Outcome: _____

Comments: _____

2. Test: _____ Outcome: _____

Comments: _____

APPENDIX A (Cont'd)

Attachments

ATTACHMENT 2. Field Test Data Sheet for Drum/Tank Sampling (cont'd)

3. Test: _____ Outcome: _____

Comments: _____

4. Test: _____ Outcome: _____

Comments: _____

5. Test: _____ Outcome: _____

Comments: _____

HAZCAT PESTICIDES KIT:

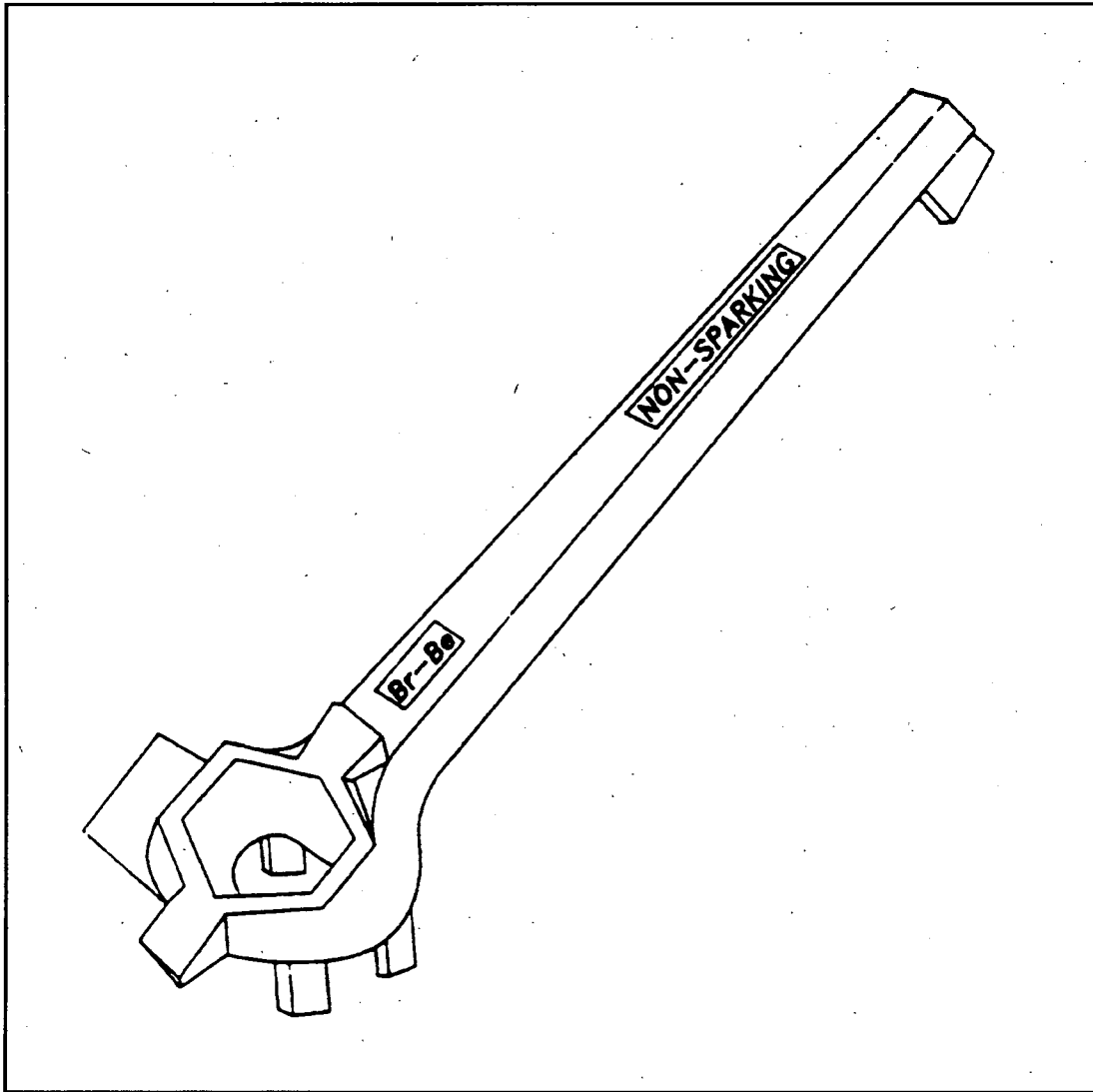
Present: _____ Not Present: _____

Comments: _____

APPENDIX B

Figures

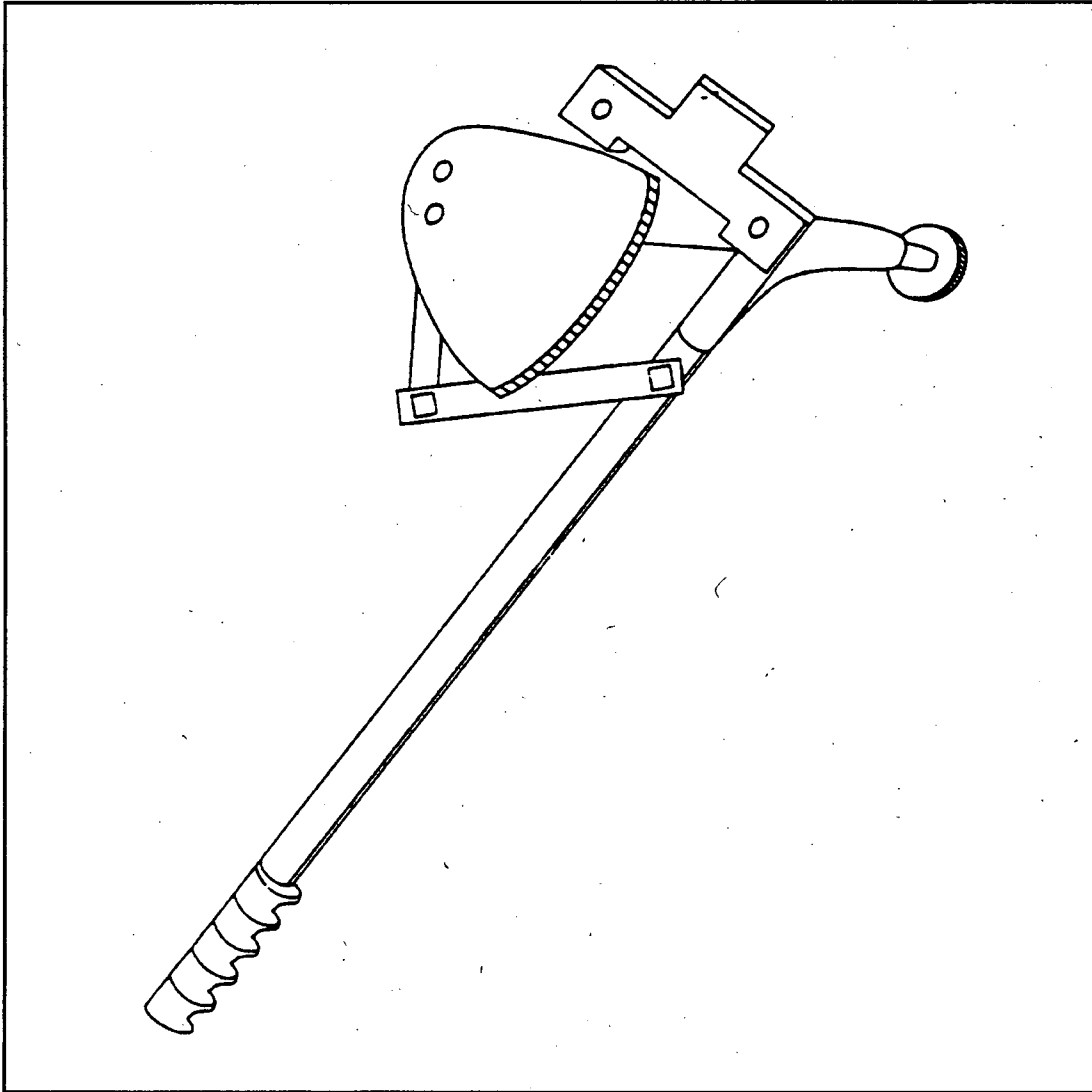
Figure 1. Universal Bung Wrench



APPENDIX B (Cont'd)

Figures

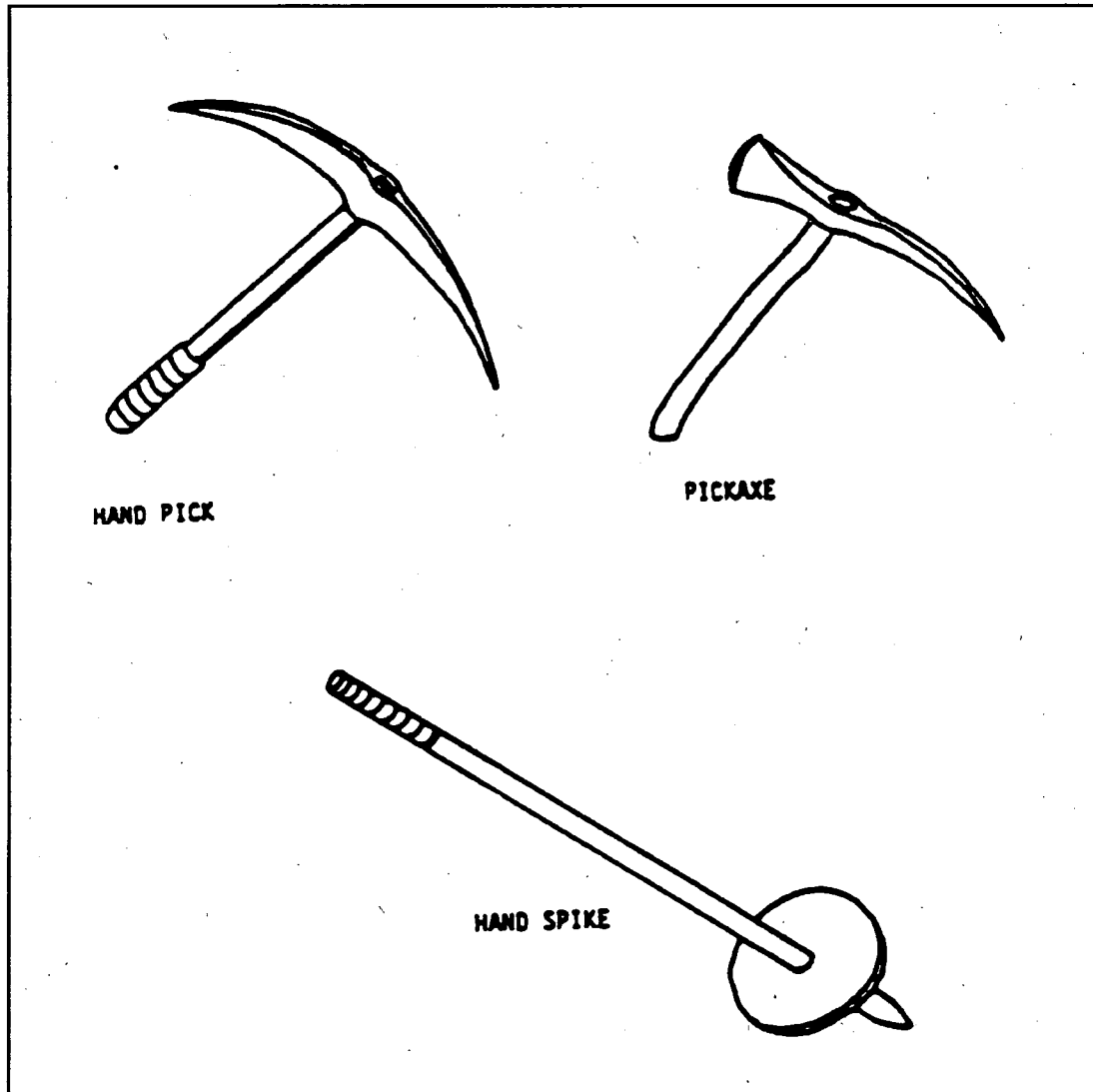
Figure 2. Drum Deheader



APPENDIX B (Cont'd)

Figures

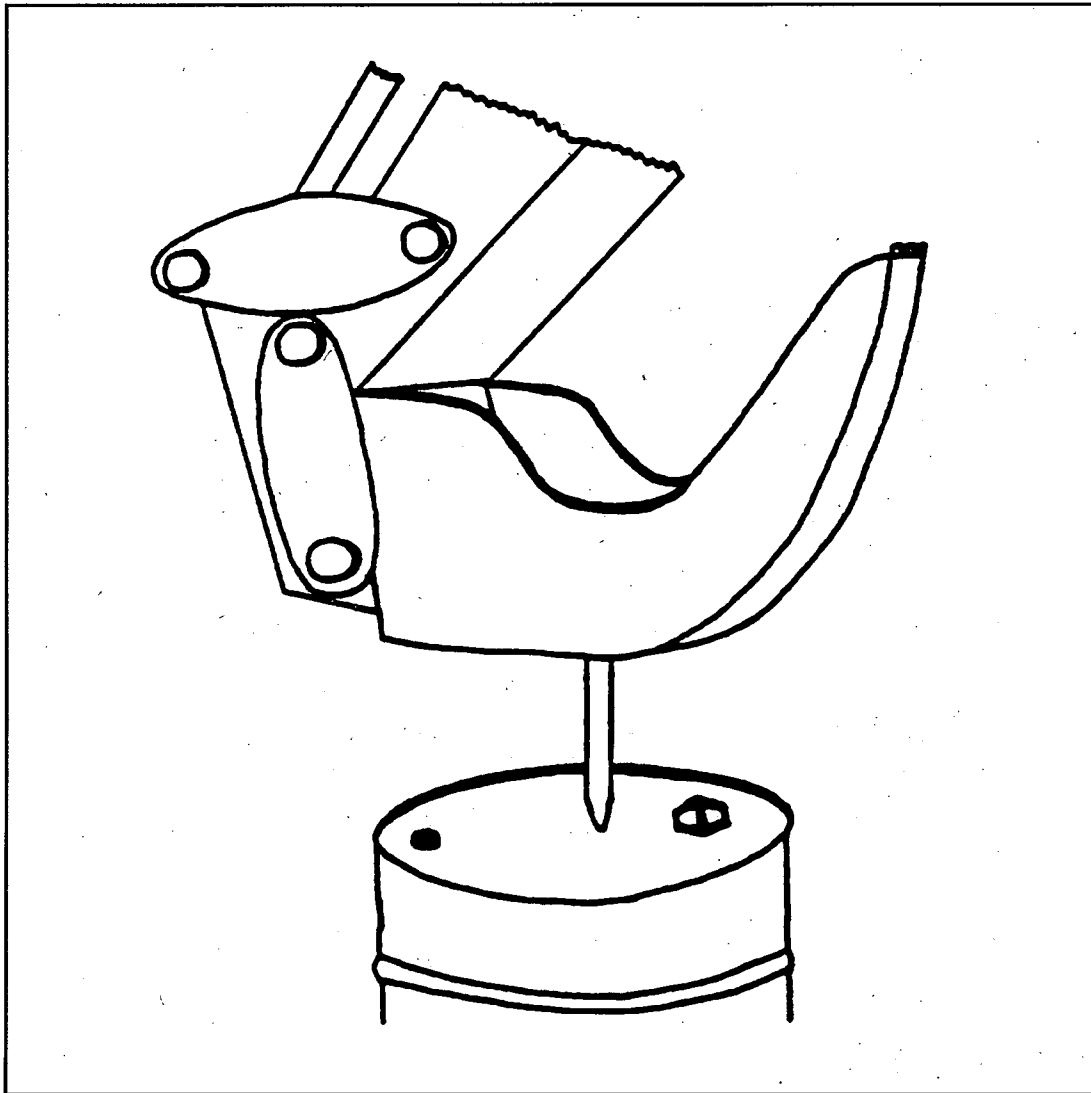
Figure 3. Hand Pick, Pickaxe, and Hand Spike



APPENDIX B (Cont'd)

Figures

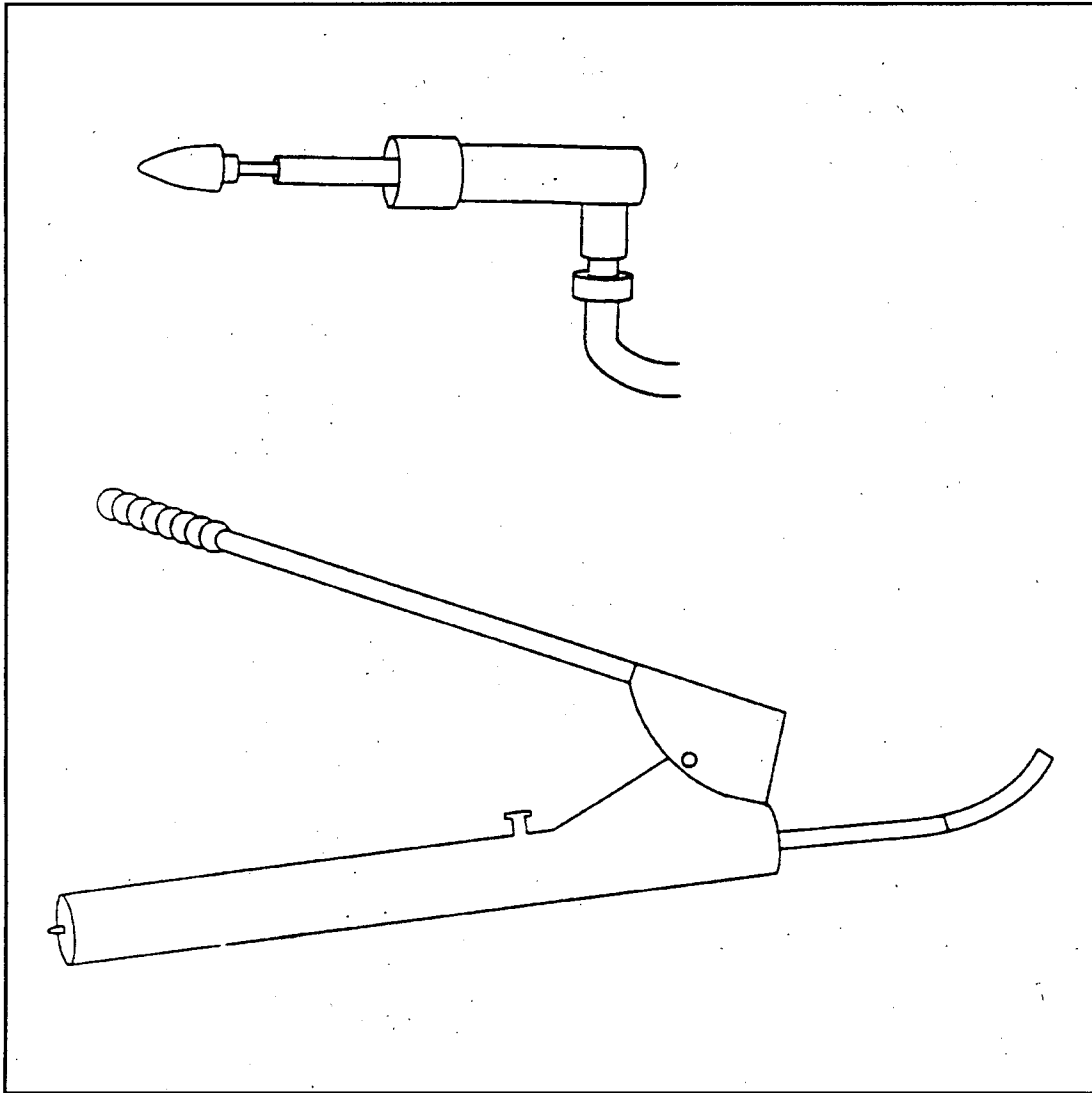
Figure 4. Backhoe Spike



APPENDIX B (Cont'd)

Figures

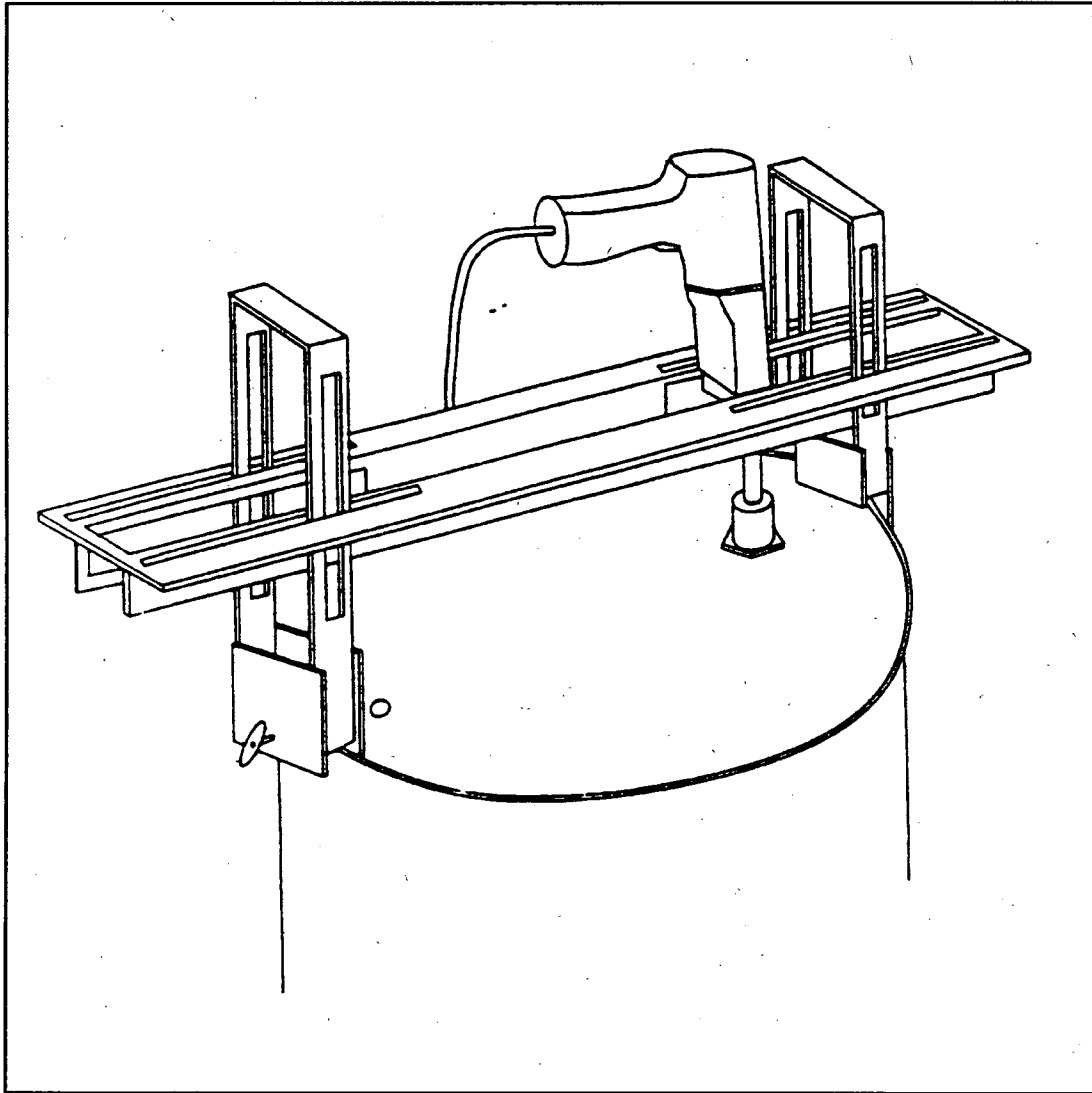
Figure 5. Hydraulic Drum Opener



APPENDIX B (Cont'd)

Figures

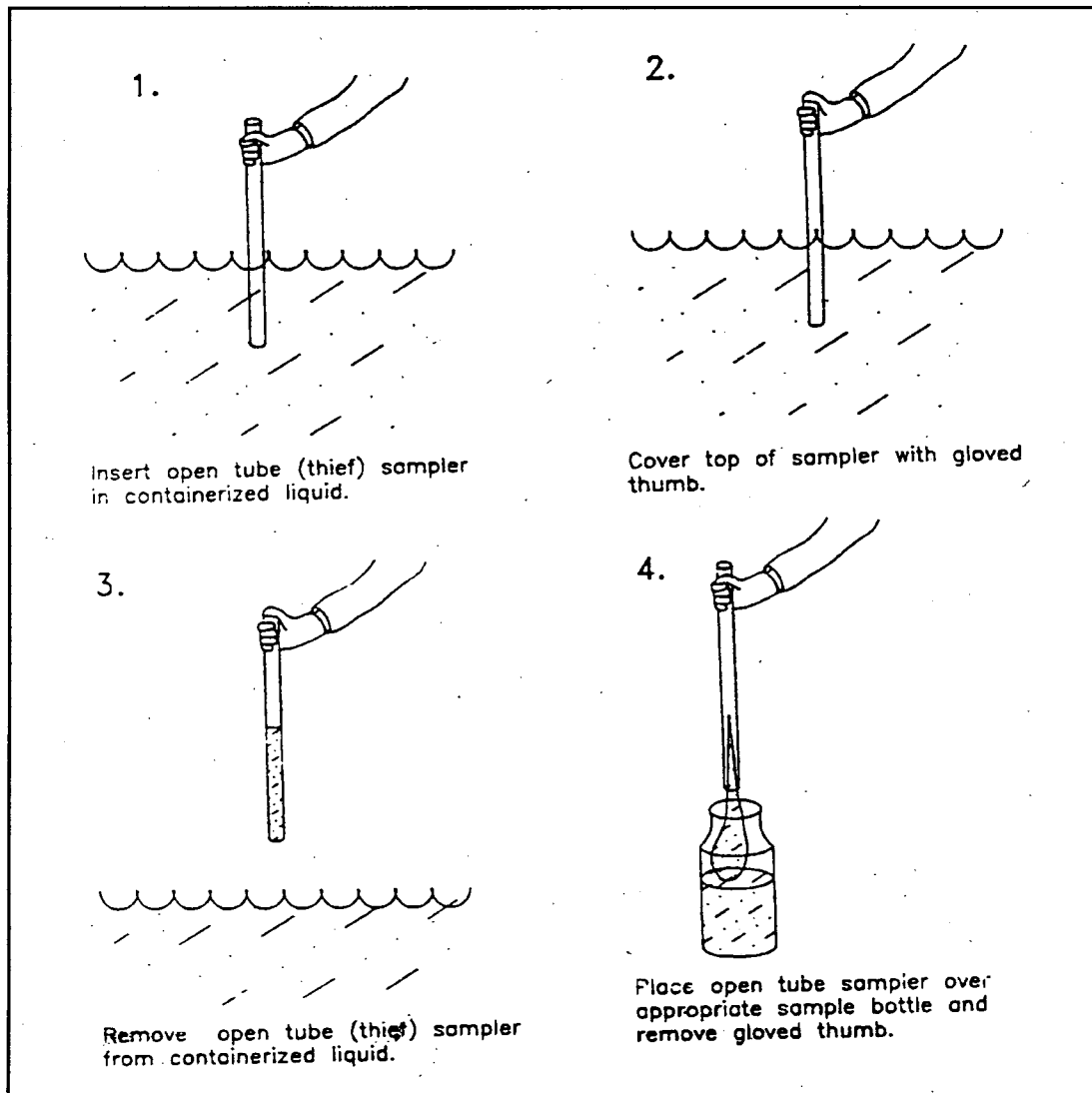
Figure 6. Pneumatic Bung Remover



APPENDIX B (Cont'd)

Figures

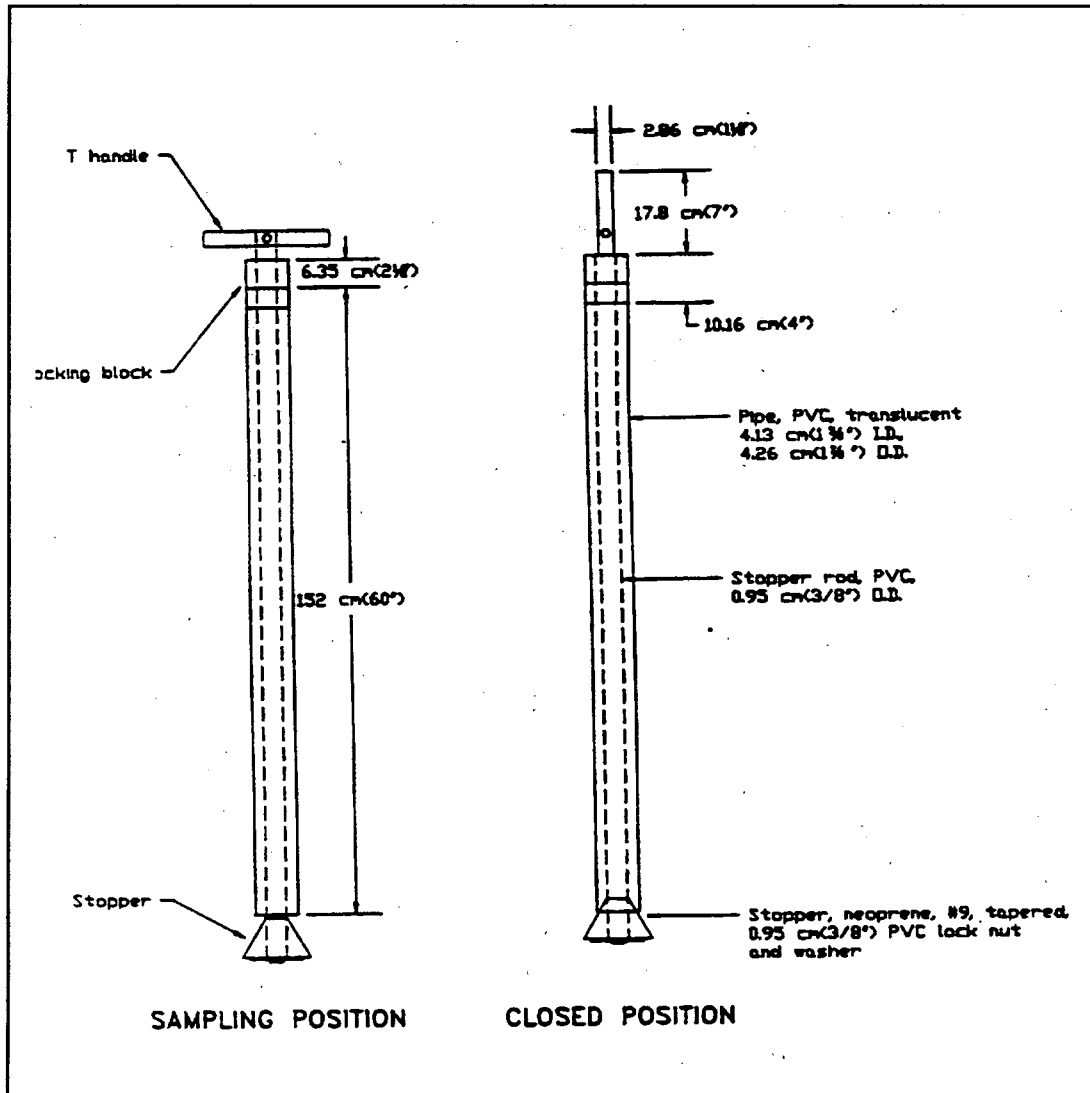
Figure 7. Glass Thief



APPENDIX B (Cont'd)

Figures

Figure 8. COLIWASA





CHIP, WIPE, AND SWEEP SAMPLING

SOP#: 2011
DATE: 11/16/94
REV. #: 0.0

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) outlines the recommended protocol and equipment for collection of representative chip, wipe, and sweep samples to monitor potential surficial contamination.

This method of sampling is appropriate for surfaces contaminated with non-volatile species of analytes (i.e., PCB, PCDD, PCDF, metals, cyanide, etc.) Detection limits are analyte specific. Sample size should be determined based upon the detection limit desired and the amount of sample requested by the analytical laboratory. Typical sample area is one square foot. However, based upon sampling location, the sample size may need modification due to area configuration.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure or other procedure limitations. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Since surface situations vary widely, no universal sampling method can be recommended. Rather, the method and implements used must be tailored to suit a specific sampling site. The sampling location should be selected based upon the potential for contamination as a result of manufacturing processes or personnel practices.

Chip sampling is appropriate for porous surfaces and is generally accomplished with either a hammer and chisel, or an electric hammer. The sampling device should be laboratory cleaned and wrapped in clean, autoclaved aluminum foil until ready for use. To

collect the sample, a measured and marked off area is chipped both horizontally and vertically to an even depth of 1/8 inch. The sample is then transferred to the proper sample container.

Wipe samples are collected from smooth surfaces to indicate surficial contamination; a sample location is measured and marked off. While wearing a new pair of surgical gloves, a sterile gauze pad is opened, and soaked with solvent. The solvent used is dependent on the surface being sampled. This pad is then stroked firmly over the sample surface, first vertically, then horizontally, to ensure complete coverage. The pad is then transferred to the sample container.

Sweep sampling is an effective method for the collection of dust or residue on porous or non-porous surfaces. To collect such a sample, an appropriate area is measured off. Then, while wearing a new pair of disposable surgical gloves, a dedicated brush is used to sweep material into a dedicated dust pan. The sample is then transferred to the proper sample container.

Samples collected by all three methods are then sent to the laboratory for analysis.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Samples should be stored out of direct sunlight to reduce photodegradation, cooled to 4°C and shipped to the laboratory performing the analysis. Appropriately sized laboratory cleaned, glass sample jars should be used for sample collection. The amount of sample required will be determined in concert with the analytical laboratory.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

This method has few significant interferences or problems. Typical problems result from rough porous

surfaces which may be difficult to wipe, chip, or sweep.

5.0 EQUIPMENT

Equipment required for performing chip, wipe, or sweep sampling is as follows:

- Lab clean sample containers of proper size and composition
- Site logbook
- Sample analysis request forms
- Chain of Custody records
- Custody seals
- Field data sheets
- Sample labels
- Disposable surgical gloves
- Sterile wrapped gauze pad (3 in. x 3 in.)
- Appropriate pesticide (HPLC) grade solvent
- Medium sized laboratory cleaned paint brush
- Medium sized laboratory cleaned chisel
- Autoclaved aluminum foil
- Camera
- Hexane (pesticide/HPLC grade)
- Iso-octane
- Distilled/deionized water

6.0 REAGENTS

Reagents are not required for preservation of chip, wipe or sweep samples. However, reagents will be utilized for decontamination of sampling equipment.

7.0 PROCEDURES

7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.
2. Obtain necessary sampling and monitoring equipment.
3. Decontaminate or preclean equipment, and ensure that it is in working order.
4. Prepare scheduling and coordinate with staff, clients, and regulatory agency, if appropriate.
5. Perform a general site survey prior to site entry in accordance with the site specific

Health and Safety Plan.

6. Mark all sampling locations. If required the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

7.2 Chip Sample Collection

Sampling of porous surfaces is generally accomplished by using a chisel and hammer or electric hammer. The sampling device should be laboratory cleaned or field decontaminated as per the Sampling Equipment Decontamination SOP. It is then wrapped in cleaned, autoclaved aluminum foil. The sampler should remain in this wrapping until it is needed. Each sampling device should be used for only one sample.

1. Choose appropriate sampling points; measure off the designated area. Photo documentation is optional.
2. Record surface area to be chipped.
3. Don a new pair of disposable surgical gloves.
4. Open a laboratory-cleaned chisel or equivalent sampling device.
5. Chip the sample area horizontally, then vertically to an even depth of approximately 1/8 inch.
6. Place the sample in an appropriately prepared sample container with a Teflon lined cap.
7. Cap the sample container, attach the label and custody seal, and place in a plastic bag. Record all pertinent data in the site logbook and on field data sheets. Complete the sampling analysis request form and chain of custody record before taking the next sample.
8. Store samples out of direct sunlight and cool to 4°C.
9. Follow proper decontamination procedures then deliver sample(s) to the laboratory for analysis.

7.3 Wipe Sample Collection

Wipe sampling is accomplished by using a sterile

gauze pad, adding a solvent in which the contaminant is most soluble, then wiping a pre-determined, pre-measured area. The sample is packaged in an amber jar to prevent photodegradation and packed in coolers for shipment to the lab. Each gauze pad is used for only one wipe sample.

1. Choose appropriate sampling points; measure off the designated area. Photo documentation is optional.
2. Record surface area to be wiped.
3. Don a new pair of disposable surgical gloves.
4. Open new sterile package of gauze pad.
5. Soak the pad with solvent of choice.
6. Wipe the marked surface area using firm strokes. Wipe vertically, then horizontally to insure complete surface coverage.
7. Place the gauze pad in an appropriately prepared sample container with a Teflon-lined cap.
8. Cap the sample container, attach the label and custody seal, and place in a plastic bag. Record all pertinent data in the site logbook and on field data sheets. Complete the sampling analysis request form and chain of custody record before taking the next sample.
9. Store samples out of direct sunlight and cool to 4°C.
10. Follow proper decontamination procedures, then deliver sample(s) to the laboratory for analysis.

7.4 Sweep Sample Collection

Sweep sampling is appropriate for bulk contamination. This procedure utilizes a dedicated, hand held sweeper brush to acquire a sample from a pre-measured area.

1. Choose appropriate sampling points; measure off the designated area. Photo documentation is optional.
2. Record the surface area to be swept.

3. Don new pair of disposable surgical gloves.
4. Sweep the measured area using a dedicated brush; collect the sample in a dedicated dust pan.
5. Transfer sample from dust pan to sample container.
6. Cap the sample container, attach the label and custody seal, and place in a plastic bag. Record all pertinent data in the site log book and on field data sheets. Complete the sampling analysis request form and chain of custody record before taking the next sample.
7. Store samples out of direct sunlight and cool to 4°C.
8. Leave contaminated sampling device in the sample material, unless decontamination is practical.
9. Follow proper decontamination procedures, then deliver sample(s) to the laboratory for analysis.

8.0 CALCULATIONS

Results are usually provided in mg/g, µg/g, mass per unit area, or other appropriate measurement. Calculations are typically done by the laboratory.

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

The following general quality assurance procedures apply:

1. All data must be documented on standard chain of custody forms, field data sheets or within the site logbook.
2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.

The following specific quality assurance activities apply to wipe samples:

For wipe samples, a blank should be collected for each sampling event. This consists of a sterile gauze pad, wet with the appropriate solvent, and placed in a prepared sample container. The blank will help identify potential introduction of contaminants via the sampling methods, the pad, solvent or sample container. Spiked wipe samples can also be collected to better assess the data being generated. These are prepared by spiking a piece of foil of known area with a standard of the analyte of choice. The solvent containing the standard is allowed to evaporate, and the foil is wiped in a manner identical to the other wipe samples.

Specific quality assurance activities for chip and sweep samples should be determined on a site specific basis.

10.0 DATA VALIDATION

A review of the quality control samples will be conducted and the data utilized to qualify the environmental results.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow EPA, OSHA and corporate health and safety procedures.

12.0 REFERENCES

U.S. EPA, A Compendium of Superfund Field Operation Methods. EPA/540/5-87/001.

NJDEP Field Sampling Procedures Manual, February, 1988.

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12.0 REFERENCES

13.0 BIBLIOGRAPHY

14.0 APPENDIX

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1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to serve as a guide to the start-up, check out, operation, calibration, and routine use of the NITON XL722S field portable x-ray fluorescence instrument for field use in screening hazardous or potentially hazardous inorganic materials. It is not intended to replace or diminish the use of the NITON 300series & 700series User's Guide. The User's Guide contains detailed information for optimizing instrument performance and for utilizing different applications.

The procedures contained herein are general operating guidelines which may be changed as required, depending on site conditions, equipment limitations, limitations imposed by Quality Assurance\Quality Control (QA\QC) procedures or other protocol limitations. In all instances, the procedures finally employed should be documented and included in the final report.

Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.1 Principles of Operation

X-ray Fluorescence (XRF) spectroscopy is a non-destructive, qualitative and quantitative analytical technique used to determine the chemical composition of samples. In a source excited XRF analysis, primary X-rays emitted from a sealed radioisotope source are utilized to irradiate samples. During interaction with samples, source X-rays may either undergo scattering (dominating process) or absorption by sample atoms in a process known as the photoelectric effect (absorption coefficient). This phenomenon originates when incident radiation knocks out an electron from the innermost shell of an atom creating a vacancy. The atom is excited and releases its surplus energy almost instantly by filling the vacancy with an electron from one of the higher energy shells. This rearrangement of electrons is associated with the emission of X-rays characteristic (in terms of energy) of the given atom. This process is referred to as emission of fluorescent X-rays (fluorescent yield). The overall efficiency of the fluorescence process is referred to as excitation efficiency and is proportional to the product of the absorption coefficient and the fluorescent yield.

1.1.1 Characteristic X-rays

The NITON XL722S utilizes characteristic X-ray lines originating from the innermost shells of the atoms: K, L, and occasionally M. The characteristic X-ray lines of the K series are the most energetic lines for any element and, therefore, are the preferred analytical lines. The K lines are always accompanied by the L and M lines of the same element. However, with energies much lower than those of the K lines, they can usually be neglected for those elements for which the K lines are analytically useful. For heavy elements such as cerium (Ce) (atomic number, Z=58), to uranium (U, Z=92), the L lines are the preferred lines for analysis. The L_{α} and L_{β} lines have almost equal intensities, and the choice of one or the other

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depends on what interfering lines might be present. A source just energetic enough to excite the L lines will not excite the K lines of the same element. The M lines will appear together with the L lines.

The NITON User's Guide contains information about the X-rays (K or L) and elements that are measured for each excitation source.

An X-ray source can excite characteristic X-rays from an element only if the source energy is greater than the absorption edge energy for the particular line group of the element (e.g., K absorption edge, L absorption edge, M absorption edge). The absorption edge energy is somewhat greater than the corresponding line energy. The K absorption edge energy is approximately the sum of the K, L, and M line energies, and the L absorption edge energy is approximately the sum of the L and M line energies of the particular element.

Energies of the characteristic fluorescent X-rays are converted (within the detector) into a train of electric pulses, the amplitudes of which are linearly proportional to the energy. An electronic multichannel analyzer measures the pulse amplitudes, which is the basis of a qualitative X-ray analysis. The number of counts at a given energy is representative of element concentration in a sample and is the basis for quantitative analysis.

1.1.2 Scattered X-rays

The source radiation is scattered from the sample by two physical processes: coherent or elastic scattering (no energy loss), and Compton or inelastic scattering (small energy loss). Thus, source backscatter (background signal) consists of two components with X-ray lines close together. The higher energy line is equal to the source energy. Since the whole sample takes part in scattering, the scattered X-rays usually yield the most intense lines in the spectrum. Furthermore, the scattered X-rays have the highest energies in the spectrum and, therefore, contribute most of the total measured intensity signal.

1.2 Sample Types

Solid and liquid samples may be analyzed with the NITON XL722S for elements potassium (K) through uranium (U) with proper X-ray source selection, application setup, measurement conditions, and instrument calibration. Typical environmental applications are:

- Heavy metals in soil (in-situ or samples collected from the surface or from bore hole drillings, etc.), sediments, and sludges
- Heavy metal air particulates collected on membrane filters, either from personnel samplers

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or from high volume samplers.

- Lead (Pb) in paint

2.0 METHOD SUMMARY

The NITON XL722S Portable XRF Analyzer employs two radioactive isotope sources: cadmium-109 (Cd-109) and americium-241 (Am-241) for the production of primary X-rays. Each source emits a specific set of primary X-rays which excite a corresponding range of elements in a sample. When more than one source can excite the element of interest, the appropriate source is selected according to its excitation efficiency for the element of interest. Each NITON XL722S analyzer will be configured with the appropriate sources depending on the applications provided with the unit.

The sample is positioned in front of the source-detector window and sample measurement is initiated, which exposes the sample to primary radiation from the source. Fluorescent and backscattered X-rays from the sample enter through the detector window and are counted in the high-performance, solid-state detector.

Elemental concentrations are computed based on ratios of analyte X-ray intensity to source backscatter. The raw ratios are corrected for spectral overlap and interelement effects utilizing correction coefficients and iteratively computed element concentrations. The NITON XL722S is factory calibrated, and the menu-driven software supports multiple calibrations called "applications." Each application is a complete analysis configuration including elements to be measured, interfering elements in the sample, and a set of calibration coefficients.

Measurement time is user controlled. Shorter measurement times (30 - 60s) are generally used for initial screening and hot spot delineation, while longer measurement times (60 - 300s) are typically used for higher precision and accuracy requirements.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

This SOP specifically describes operating procedures for the NITON XL722S; hence, this section is not applicable to this SOP.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

The total method error for XRF analysis is defined as the square root of the sum of squares of both instrument precision and user or application related error. Generally, the instrument precision is the least significant source of error in XRF analysis. User or application related error is generally more significant and will vary with each site and method used. The components of the user or application related error are the following.

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4.1 Sample Placement

This is a potential source of error because the X-ray signal decreases as the distance from the radioactive source is increased. However, this error is minimized by maintaining the same distance for each sample. Sample geometry with respect to the source/detector is also important. A tilted sample may cause analytical error. The NITON XL722S ratios analyte X-ray lines to source backscatter, which minimizes this type of error.

4.2 Sample Representativity

In order to accurately characterize site conditions, samples collected must be representative of the site or area under investigation. Representative soil sampling ensures that a sample or group of samples accurately reflects the concentration of the contaminant(s) of concern at a given time and location. Analytical results from representative samples reflect the variation in pollutant presence and concentration range throughout a site. Variables affecting sample representativeness include: (1) geologic variability, (2) contaminant concentration variability, (3) collection and preparation variability, and (4) analytical variability. Attempts should be made to minimize these sources of variability. For additional information on representative sampling, refer to the "Removal Program Representative Sampling Guidance, Volume 1 - Soil."⁽¹⁾

4.3 Reference Analysis

Soil chemical and physical matrix effects may be corrected (to some extent) by adjusting XRF results (via regression) using site-specific soil samples which have been analyzed by Inductively-Coupled Plasma (ICP) or Atomic Absorption (AA) spectroscopy methods. A major source of error can result if these samples are not representative of the site and/or if the analytical error is large. Additionally, when comparing XRF results with reference analyses results, the efficiency of the sample digestion reference analysis should be considered. Some digestion methods may breakdown different sample matrices more efficiently than others.

4.4 Chemical Matrix Effects

Chemical matrix effects result from differences in concentrations of interfering elements. These effects appear as either spectral interferences (peak overlaps) or as X-ray absorption/enhancement phenomena. Both effects are common in soils contaminated with heavy metals. For example, iron (Fe) tends to absorb copper (Cu) X-rays, reducing the intensity of Cu measured by the detector. This effect can be corrected mathematically through the use of interelement correction coefficients.

4.5 Physical Matrix Effects

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Physical matrix effects are the result of variations in the physical character of the sample. They include parameters such as particle size, uniformity, homogeneity, and surface condition. For example, consider a sample in which the analyte exists in the form of very fine particles within a matrix composed of much coarser material. If two separate aliquots of the sample are prepared in such a way that the matrix particles in one are much larger than in the other, then the relative volume of analyte occupied by the analyte-containing particles will be different in each. When measured, a larger amount of the analyte will be exposed to the source X-rays in the sample containing finer matrix particles; this results in a higher intensity reading for that sample and, consequently, an apparently higher measured concentration for that element.

4.6 Application Error

Generally, the error in the application calibration model is insignificant (relative to the other sources of error) **PROVIDED** the instrument's operating instructions are followed correctly. However, if the sample matrix varies significantly from the design of the application, the error may become significant (e.g., using the Bulk Sample [soils] application to analyze a 50 percent iron mine tailing sample).

4.7 Moisture Content

Sample moisture content affects the analytical accuracy of soils or sludges. The overall error may be secondary when the moisture range is small (5-20 percent), or it may be a major source of error when measuring the surface of soils that are saturated with water. (NOTE: attempting an in-situ measurement on a saturated soil may damage the instrument.)

4.8 Cases of Severe X-ray Spectrum Overlaps

When present in the sample, certain X-ray lines from different elements can be very close in energy and, therefore, can interfere by producing a severely overlapped spectrum.

Typical spectral overlaps are caused by the K_{β} line of element Z-1 (or as with heavier elements, Z-2 or Z-3) overlapping with the K_{α} line of element Z. This is the so-called K_{α}/K_{β} interference. Since the $K_{\alpha}:K_{\beta}$ intensity ratio for the given element usually varies from 5:1 to 7:1, the interfering element, Z-1, must be present in large concentrations in order to affect the measurement of analyte Z. For example, the presence of large concentrations of iron (Fe) could affect the measurement of cobalt (Co). The Fe K_{α} and K_{β} energies are 6.40 and 7.06 KeV, respectively. The Co K_{α} energy is 6.93 KeV. The resolution of the detector is approximately 300 eV. Therefore, large amounts of Fe in a sample will result in spectral overlap of the Fe K_{β} with the Co K_{α} peak (see Figure 1, Appendix A) and the resultant X-ray spectrum will include TOTAL counts for Fe plus Co lines.

Other interferences arise from K/L, K/M, and L/M line overlaps. While these are less common, the

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following are examples of severe overlap:

As K_{α} /Pb L_{α} , Ti K_{α} /Ba L_{α}

In the arsenic (As)/lead case, Pb can be measured from the Pb L_{α} line, and arsenic from either the As K_{α} or the As K_{β} line; this way the unwanted interference can be corrected. However, due to the limits of mathematical corrections, measurement sensitivity is reduced. Generally, arsenic concentrations can not be efficiently calculated in samples with Pb:As ratios of 10:1 or more. This may result in zero arsenic being reported regardless of what the actual concentration is.

The NITON XL722S uses overlap factors to correct for X-ray spectral overlaps for the elements of interest for a given application.

5.0 EQUIPMENT / APPARATUS

5.1 Description of the NITON XL722S System

The NITON XL722S is a complete, hand-held, portable XRF analyzer weighing less than three pounds. It utilizes the method of Energy Dispersive X-Ray Fluorescence (EDXRF) spectroscopy to determine the elemental composition of soils, sludges, particulate, paint, and other waste materials.

The NITON XL722S analyzer includes two compact, sealed radiation sources: Cd-109 and Am-241. The user selects the source and the analyzer software reports concentrations based on stored information for each application. Measurement time is user determined. The NITON XL722S utilizes a high performance, electrically-cooled, solid-state detector optimized for L-shell and K-shell X-ray detection.

The unit provides internal non-volatile memory for storage of 1000 bulk and/or thin sample spectra and multi-element analysis reports, or up to 3000 paint-mode test results. A RS-232 serial port is provided for downloading results and spectra to a PC. The multi-element analysis reports and spectra can be displayed on the instrument's display screen. The replaceable and rechargeable Nickel Metal Hydride battery pack provides for field-portable operation.

The NITON XL722S is supplied with one or more applications. The "Bulk Sample" (soil samples) application is for analysis of up to 25 metals, where the balance of the sample (that portion not directly measured by the instrument) is essentially silica (SiO_2). The "Thin Sample" application is for analysis of thin films such as air monitoring filters or wipes. The "Lead-Based Paint" application is for analyzing Pb in paint films. NITON LLC will also develop new applications to meet user specific requirements (e.g., adding elements to the "Soil Samples" application).

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The NITON XL722S is powered from its 8-hour capacity battery. The manufacturer's operating condition specifications are: Temperature range 20 to 120° Fahrenheit (F); Humidity range 0 to 95% RH.

5.2 Equipment and Apparatus List

5.2.1 NITON XL722S Analyzer System

The complete NITON XL722S Analyzer System includes:

- Hand-held analyzer unit for data acquisition, processing, and display. Includes: high-performance, solid-state detector, two excitation sources (Cd-109, Am-241), data processing software, and control panel/results display.
- RS-232C Serial I/O Interface cable
- Silicon dioxide (SiO₂) blank check sample
- Three NIST soil SRMs: 2709, 2710, and 2711, and one RCRA check sample
- Battery charger
- Two battery packs
- System carrying/shipping case and field carrying case/holster
- Soil sample analysis/preparation accessories in separate carrying/shipping case
- NITON XL722S User's Guide and NITON Xtras PC utilities software.

5.2.2 Optional Items

- 31-mm diameter sample cups
- XRF polypropylene film, 0.2 mil thick
- Windows 2000 based Personal Computer (PC)
- Spare battery packs and spare charger

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See the NITON XL722S Accessories List for additional options.

For mobile/lab or laboratory X-ray sample preparation accessories (such as drying ovens, grinders, sieves, etc.), consult general laboratory equipment suppliers.

5.3 Peripheral Devices

The NITON XL722S may be used with a PC to download results/spectra, and for customized reports.

5.3.1 Communication Cable Connection

Plug the round end of the RS-232 Serial I/O cable into the NITON XL722S connector (the connection left of the on/off/reset switch) and the 9-pin connector of the cable into the serial port of the PC.

5.3.2 NITON Xtras 5.7e Software

The PC must be running the NITON XTras v5.7e software to communicate with the NITON XL722S. The XTras software allows you to select various configurations for downloading, exporting, displaying, and reporting results/spectra. Refer to the NITON User's Guide and XTras Quickstart Guide for details.

6.0 REAGENTS

Generally, soil calibration standards are not necessary for site screening and extent of contamination analyses with the NITON XL722S. The unit's performance can be verified by analyzing the NIST soil SRMs provided with the unit. Refer to the NITON User's Guide for chemical composition of the NIST SRMs.

7.0 PROCEDURE

7.1 Operation

Refer to the NITON User's Guide for detailed instrument operating procedures and screen illustrations.

7.1.1 Startup

To remove the battery, loosen the two screws on the end of the unit below the on/off/reset switch and gently lift the battery pack away from the connector on the unit's base. Insert a fresh battery pack and tighten the set screws.

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Apply power to the NITON by sliding the on/off/reset switch to the on position. Sometimes the instrument's battery saving features momentarily delay startup. If the NITON does not turn on immediately, turn it off, wait a few seconds, and turn it on again. Each time the NITON is turned on, the Main Menu appears and the screen arrow points to **Calibrate & Test**.

Allow the NITON to warm up for a minimum of 15 minutes after it has been turned on before performing analysis.

7.1.2 Precautions

The NITON XL722S should be handled in accordance with the following radiological control practices.

Refer to the NITON User's Guide for detailed discussion of Radiation Safety practices.

1. The NITON XL722S should always be in contact with the surface of the material being analyzed, and that material should completely cover the aperture when the sources are exposed. Do not remove a sample or move the unit while the shutter is open.
2. When the sources are exposed, under no circumstances should the NITON XL722S be pointed at the operator or surrounding personnel.
3. Do not place any part of the operator's or co-worker's bodies in line of exposure when the sources are exposed or partially covered.
4. The shutter must be closed with the shutter safety lock engaged when not in use.
5. The manufacturer (NITON LLC) must be notified immediately of any condition or concern relative to the NITON XL722S's structural integrity, source shielding, source switching condition, or operability.
6. The appropriate state agency or the Nuclear Regulatory Commission (NRC) office must be notified immediately of any damage to the radioactive source, or any loss or theft of the device (see factory supplied data on radiological safety).
7. Labels or instructions on the NITON XL722S(s) must not be altered or removed.
8. The user must not attempt to open the unit.

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9. The source(s) in the unit must be wipe-tested every 6 months as described in the NITON XL722S User's Guide. The leak test certificates must be kept on file, and a copy must accompany the instrument at all times.
10. The bulk test platform or equivalent sample stage provided by NITON LLC must be used whenever the NITON XL722S is used for measuring samples contained in cups.
11. The NITON XL722S should not be dropped or exposed to conditions of excessive shock or vibration.

Additional precautions include:

1. The NITON XL722S should always be stored in it's waterproof, drop-proof carrying case.
2. The battery charging unit should only be used in dry conditions.
3. Battery packs should be changed only in dry conditions.

7.2 Control Panel and Menu Software

This section outlines the control panel buttons and basic menu software. Detailed illustrations of the control panel and screen displays are in the NITON User's Guide.

7.2.1 Control Panel Buttons

The NITON control panel consists of three buttons; Clear/Enter, right arrow (->), and left arrow (<-). These buttons allow the operator to navigate all the NITON screens and menus. The amount of time that the button is held down also controls the function of the buttons. Pressing the Clear/Enter button briefly (less than 1 second) or pressing the right (->) or left (<-) arrow buttons scrolls through the listed items shown on the screen. Holding down the Clear/Enter button for a longer period (more than 3 seconds) activates a different screen.

7.2.2 The Setup Menu

The Setup Menu is used to check instrument specifications, to set date and time, to illuminate the screen continuously, or to select a different testing mode. Once set up, this screen will remain the same each time the NITON is turned on until it is reset. To activate,

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select Setup Menu from the Main Menu with the arrow buttons and press the Clear/Enter button to enter the Setup Menu. Refer to the NITON User's Guide for detailed instructions on using the Setup Menu.

7.2.3 Calibrate & Test

When the screen arrow (->) is on Calibrate & Test, press the Clear/Enter button to start the self-calibration process. This process calibrates detector energy gain/zero so that analyte X-rays are in their proper spectral location. Self-calibration takes about one to two minutes. When it is complete, the instrument will beep and the Ready to Test screen will appear. The self-calibration process should be performed every 1-2 hours during sample analysis to maintain proper detector calibration.

7.2.4 The Ready to Test Screen

This screen displays: the current date and time, the instrument serial number, the indication that the instrument is ready to test, the testing mode, the action level for "positive" or "negative" determination of lead in paint (lead based paint application only), the detector energy resolution, and the source strength.

CAUTION: Check the date and time. If they are not correct, reset them before taking any measurements (see NITON User's Guide). Readings will not be accurate unless date and time are correct.

7.2.5 The Measurement Screen

The highest concentration elements are displayed in ppm (with the two-sigma confidence intervals) on the first measurement screen. The test time is also displayed.

7.2.6 The Summary Screen

When the operator ends a reading, the Measurement Screen is replaced by the Summary Screen. Results are displayed for 14 elements on NITON XL700 series models. These are divided into two groups: detected elements, and elements that were not detected. Press the arrow buttons to scroll through the element list. An element is classified detected when the measured concentration (ppm) is at least 1.5-times the confidence interval (ie, 3-sigma). Detected elements are displayed as in the Measurement Screen. Non-detected elements are shown as "< xx", where xx is the three-sigma instrument detection limit for that sample. The instrument detection limit (3-sigma) for each element is calculated for each sample.

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7.3 Preoperational Checks

7.3.1 Energy Calibration

An energy calibration (Calibrate & Test) should be performed as required to ensure proper energy calibration (eg, after an instrument is shipped). The Calibrate & Test function is located in the Main Menu (see section 7.2.3).

7.3.2 Resolution Check

The resolution check examines the detector's ability to resolve X-ray energies. This should be performed once at the beginning of the day. Record/document the Energy Resolution in the Ready to Test screen (after Calibrate & Test). The value should not vary significantly from day to day and should typically be less than 400 eV. If the unit fails to meet this specification, call NITON LLC for assistance.

7.3.3 Blank (Zero) Sample Check

The blank (Zero) sample check is performed to monitor the instrument's zero drift in the selected application. The blank sample check only applies to the application (test mode) currently selected. This should be done once at the beginning of the day, after Calibrate & Test, after selecting a test mode, and whenever the instrument exhibits a persistent drift on a blank or low-level sample.

Load the SiO₂ Blank (supplied with the NITON unit) in the NITON Bulk Sample Test Platform. Analyze for 60 seconds (source seconds) with each source in the unit. Review results. All elemental results should be reported as non-detected (<xx, where xx is the 3-sigma instrument detection limit). Repeat the measurement if the unit fails to meet these specifications. If several elements continue to be significantly out of these specifications, check the plastic window and the blank sample for contamination. Perform the blank (Zero) sample check again. Save the results/spectra for documentation.

7.3.4 Target Element Response Check

The purpose of the target element response check is to ensure that the instrument and the selected application are working properly prior to performing sample analysis. This check should be performed at the beginning of the day. Use the NIST SRM 2709, 2710, and 2711 standards provided with the NITON unit to check the Soil Samples application. These samples should be measured using the same source acquisition times that will be used for sample analysis. Save the sample check results/spectra for documentation.



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7.4 Source Measuring Time

The source measuring time is user controlled. Generally, the element detection limit is reduced by 50 percent for every four-fold (x4) increase in source measuring time. Although counting statistics improve as measurement time increases, the practical upper limit for typical applications is about 300 seconds.

The NITON XL700 units measure time in "source seconds". This includes an automatic correction for source decay so that 60 source seconds will have a constant precision regardless of source age. The correction extends measurement time to correct for source strength lost through the decay process.

A minimum measuring time of 60 source seconds for each source is recommended when using the Soil Samples application. Measuring times for a source that excites a target element can be increased if lower detection limits are required.

7.5 Sample Handling and Presentation

When making XRF measurements, be sure to maintain constant measurement geometry in order to minimize variations in analysis results. Document any anomalies in measurement geometry, sample surface morphology, moisture content, sample grain size, and matrix (see Section 4.0).

7.5.1 Soil Samples

Soil samples may be analyzed either in-situ or in XRF sample cups (after preparation). The Soil Samples application assumes the sample to be infinitely thick. For in-situ measurements this is the case, however, for sample cup measurements it is advisable to fill the cup nearly full and use the supplied paper disk and cotton ball to hold the sample firmly against the sample cup window. This ensures that the sample is as uniformly thick as possible from analysis to analysis. The NITON XL722S bulk sample test platform or equivalent bulk sample platform provided by NITON must be used when analyzing sample cups.

An area for in-situ analysis should be prepared by removing large rocks and debris. The soil surface should be flat and compact prior to analysis. The NITON XL722S should be placed in its in-situ adaptor and held firmly on the ground to maximize contact with the ground. The unit should not be moved during analysis. Analysis of water saturated soils should be avoided. Use of varying thicknesses of plastic (bags) have been shown to interfere with light element (low atomic number) measurements and may affect the calculation of the other element concentrations.⁽²⁾ Additionally, plastic may contain significant levels of target element contamination. The NITON XL722S ratios analyte line intensity to that from source

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backscatter and, therefore, may be less susceptible to bag thickness effects. Refer to the NITON User's Guide for details on analyzing Soil Samples (Bulk Samples).

~~Course-grained soil conditions or nuggets of contaminated material may preclude a truly representative sample and adversely affect the analysis results (typically by under reporting the target element). Such samples should be prepared before analysis. Preparation consistency is important to minimize variation in analytical results.~~

This application is designed for soil with the assumption that the balance of the material is essentially silica. If samples with a much lighter (lower atomic number) balance are analyzed, the results may be elevated by a factor of two to four. Contact NITON LLC for help in analysis of different matrices.

7.5.2 Thin (Filter) Samples

The Thin Samples application is for analysis of thin samples such as particulates on filters or wipes. The detection limits are affected by the thickness of the substrate. Best results are obtained on the thinnest substrates. Always use the Dust wipe and Filter Test platform when measuring thin samples. This is not only for user safety, but also ensures a controlled environment to facilitate testing thin samples. ~~Contaminated material captured on filters or wipes is not usually deposited uniformly. Therefore, to produce meaningful results, several readings must be taken for each thin sample measurement.~~ The average or sum of these readings is the reported value for the measurement. Refer to the NITON User's Guide for details on analyzing Thin Samples.

7.5.3 Lead in Paint

To analyze for lead in paint on a surface (eg, wall, counter, etc.), the area selected for analysis should be smooth, representative and free of surface dirt. The NITON XL722S should be held firmly on the surface to maximize contact. The probe should not be moved during analysis. Refer to the NITON User's Guide for details on analyzing lead paint samples.

7.6 Downloading Stored Results and Spectra

Results (analytical reports) and spectra which have been stored in the NITON XL722S internal memory should be downloaded and captured in disk files on a PC (see section 5). NITON LLC provides software (Xtras 5.7e) for this purpose. Additionally, results or spectra may be exported to text files for importing into a spreadsheet. Refer to the instructions provided with the programs for details on their operation.

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After capturing results to a file, print a copy and save both the disk files and the printout for future reference and documentation purposes.

7.7 Instrument Maintenance

NOTE: All service except exterior cleaning must be performed by NITON LLC. Do not attempt to make repairs yourself. Opening the case of the NITON will void the Warranty.

7.7.1 Exterior Cleaning

When the Kapton plastic window on the bottom of the instrument becomes dirty, the performance of the NITON unit will be affected. ~~Clean the window gently with cotton swabs. Clean the instrument's metal case with a soft cloth. Never use water, detergents, or solvents. These may damage the instrument.~~

7.7.2 Further Information and Troubleshooting

Refer to the NITON XL722S User's Guide for additional detailed operational and/or maintenance and troubleshooting instructions. If no solution is found in the manual, contact NITON LLC for assistance.

An instrument log should be maintained to document specific corrective actions taken to alleviate any instrumental problems, or for recording any service that has been performed.

8.0 CALCULATIONS

The NITON XL722S is a direct readout instrument that does not require any external calculations.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

9.1 Precision

The precision of the method is monitored by reading a low- or mid-target element concentration sample (eg, a SRM or the NITON RCRA sample) at the start and end of sample analysis and after approximately every tenth sample. Determining the precision around the site action level can be extremely important if the XRF results are to be used in an enforcement action. Therefore, selection of a sample with a target element concentration at or near the site action level or level of concern is recommended. The sample is analyzed by the instrument for the normal field analysis time, and the results are recorded. A minimum of seven measurements should be made during field activities. The standard deviation for each target element is calculated. The relative standard deviation (RSD) of the sample mean can be used to calculate precision. The RSD should be within ± 20 percent.⁽³⁾

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9.2 The Method Detection Limit (MDL) and Method Quantitation Limit (MQL)

The MDL and MQL may be calculated from the measurement of either a low or blank sample (or a SRM) at the start and end of sample analysis, and after approximately every tenth sample. Alternatively, the SiO₂ blank or "clean" sand may be used if a blank soil or sediment sample is unavailable.

Measure the MDL sample using the same application and measuring time used for routine samples. A minimum of seven measurements should be made during field activities. Calculate the sample standard deviation of the mean for each target element, and round up to the next whole number prior to calculating the MDL and MQL.

The definition of the MDL is three times the calculated standard deviation value. The definition of the MQL is 10 times the calculated standard deviation value.

9.3 Reporting Results

All raw XRF data should be recorded including the individual results of multiple analyses of samples and sampling points. The average and concentration range of each multiple analysis should also be reported.

A "reported" value for each analysis or average of multiple analyses should be processed in the following manner.

1. Round the value to the same degree of significance contained in the calibration or check standard sample assay values (usually two). Round to 2 significant figures for sample results. DO NOT round results for standards used to determine MDL or RSD values (use raw data).
2. Report all values less than the MDL as not detected (U).
3. OPTIONAL: Flag and note all values greater than or equal to the MDL and less than the MQL (usually with a "J" next to the reported value).
4. Report all values equal to or greater than the MDL and within the linear calibration range.

9.4 Accuracy

Accuracy, relative to a specific digestion method and elemental analysis procedure, is determined by submitting a sample analyzed by XRF methods (prepared sample cups may be submitted) for confirmatory AA or ICP analysis at a laboratory.

The on-site analysis of soils by portable XRF instrumentation should be considered a screening effort

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only (DC1 data). Data derived from the instrument should be used with discretion. Confirmatory analyses on a subset of the screening samples (minimum 10 percent) can be used to determine if the XRF data meets DC2 data objectives. The confirmation samples should ideally be selected randomly from the sample set and include a number of samples at or near the critical level. The results of the laboratory analysis (dependent) and the XRF analysis (independent) are evaluated with a regression analysis. The coefficient of determination (r^2) should be 0.7 or greater.⁽³⁾

Correcting the XRF results based on confirmatory analyses should only be undertaken after careful consideration. The confirmatory analysis (AA or ICP) is an estimate of the concentration of metal contamination and is dependent upon the digestion method and sampling methodology used. Since XRF is a total elemental technique, any comparison with referee results must account for the possibility of variable extraction efficiency, dependent upon the digestion method used and its ability to dissolve the waste or mineral form in question.

9.5 Matrix Considerations

Other types of QA/QC verification should include verification that the instrument calibration is appropriate for the specific site to be assessed. This includes verification of potential multiple soil matrix types that may exist at a site. Matrix differences which affect the XRF measurement include large variations in calcium content, which may be encountered when going from siliceous to calcareous soils, as well as large variations in iron content.

10.0 DATA VALIDATION

10.1 Confirmation Samples

Confirmation samples are recommended at a minimum rate of 10 percent and are required if DC2 data objectives have been established for site activities.⁽³⁾ Ideally, the sample cup that was analyzed by XRF should be the same sample that is submitted for AA/ICP analysis. When confirming an in-situ analysis, collect a sample from a 6-inch by 6-inch area for both an XRF measurement and confirmation analysis.

The XRF and confirmatory AA/ICP results are analyzed with a regression analysis using a statistical program (such as SAS®) or a spreadsheet with the intercept calculated in the regression. The coefficient of determination (r^2) between XRF and AA/ICP data must be 0.7 or greater for DC2 data objectives.⁽³⁾

10.2 Recording Results

Record all results and monitoring activities in a laboratory or field notebook. Also, results may be recorded electronically on a hard drive or floppy disk.

11.0 HEALTH AND SAFETY

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When working with potentially hazardous materials, follow U.S. EPA, OSHA, corporate and/or any other applicable health and safety practices.

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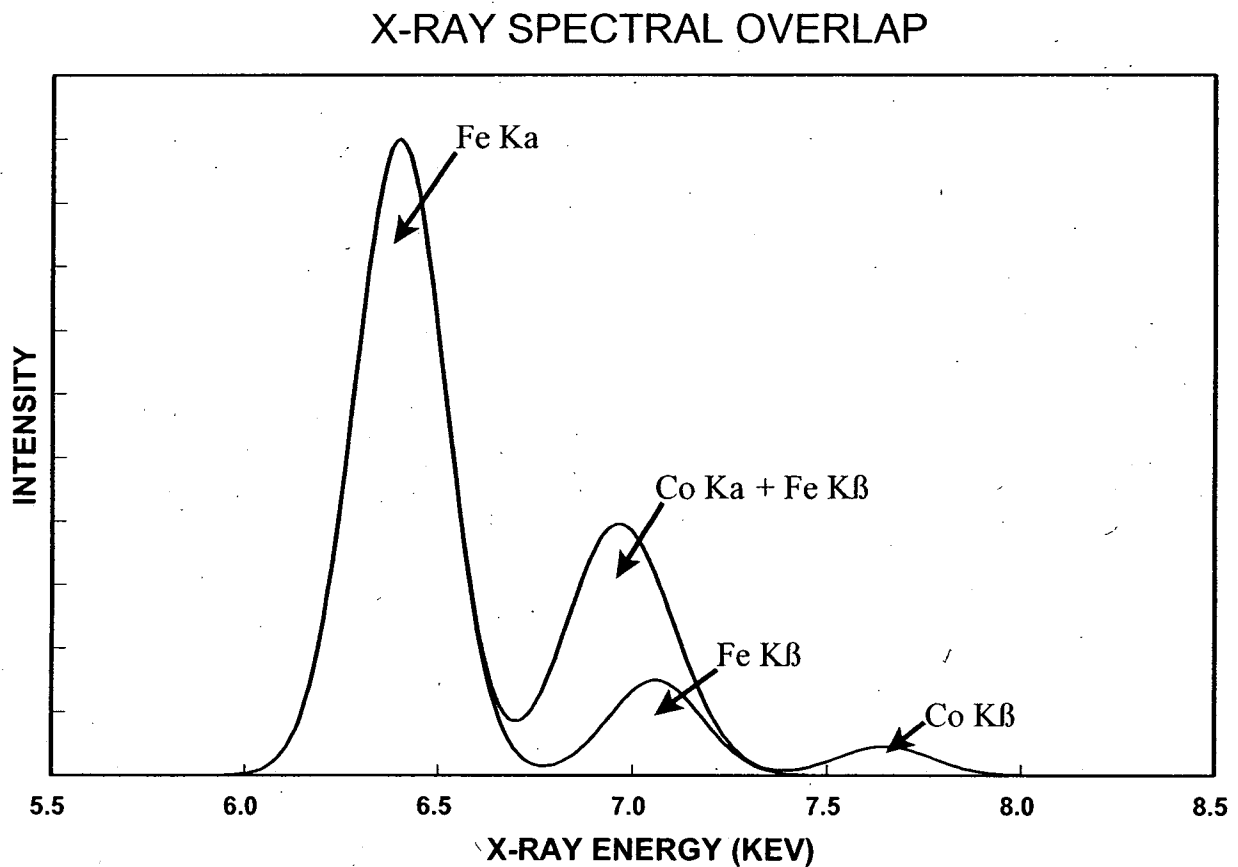
DRAFT
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Figures
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FIGURE 1. X-Ray Spectral Plot Showing Overlap of Iron K_{β} X-Rays in the Cobalt K_{α} Measurement Region.



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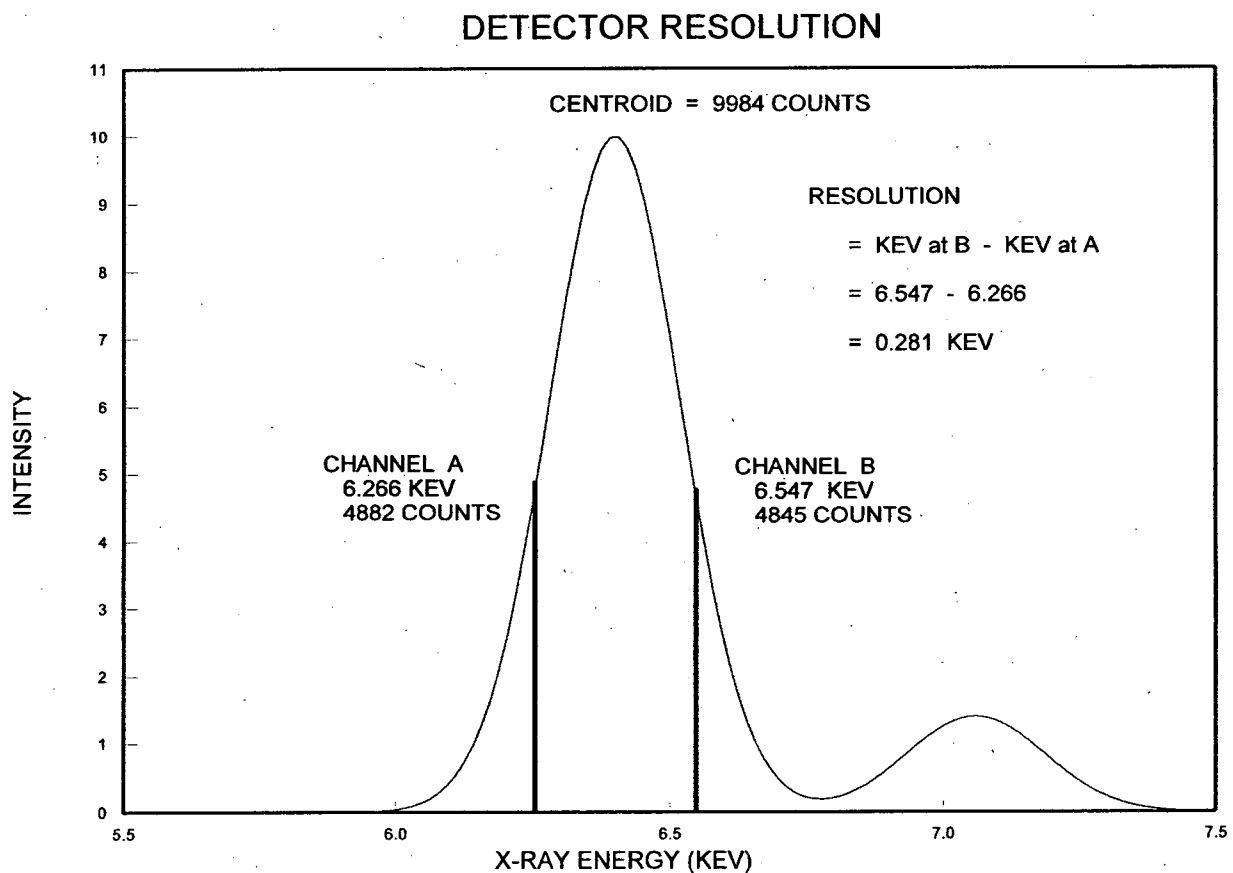
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FIGURE 2. Iron X-Ray Spectrum Illustrating Detector Resolution



ATTACHMENT C:

Drum Inventory & Field Testing Log

Drum Location Log

Air Monitoring Work Sheet

DRUM INVENTORY & FIELD TESTING LOG

SITE NAME: _____ SAMPLE #: _____ DRUM NUMBER: _____

LOGGER: _____ SAMPLER: _____ DATE/TIME: _____

DRUM DESCRIPTION:

CONSTRUCTION		TYPE		CONDITION		
Fiber <input type="checkbox"/>	Poly <input type="checkbox"/>	Poly Lined <input type="checkbox"/>	"Overpack" <input type="checkbox"/>	rusted <input type="checkbox"/>	leaking <input type="checkbox"/>	dented <input type="checkbox"/>
Steel <input type="checkbox"/>	Nickel <input type="checkbox"/>	Open Top <input type="checkbox"/>	Ring Top <input type="checkbox"/>	bulging <input type="checkbox"/>	perforated <input type="checkbox"/>	good <input type="checkbox"/>
Stainless Steel <input type="checkbox"/>	Other <input type="checkbox"/>	Closed Top <input type="checkbox"/>		other _____		

DRUM SIZE (Gallons): 85 <input type="checkbox"/>	55 <input type="checkbox"/>	42 <input type="checkbox"/>	30 <input type="checkbox"/>	15 <input type="checkbox"/>	10 <input type="checkbox"/>	5 <input type="checkbox"/>	Other _____
MFG NAME							
CHEMICAL NAME							
DRUM MARKINGS							
DRUM LABELS							

FIELD AIR MONITORING INSTRUMENT READINGS: MultiRAE _____ PID _____ FID _____ RAD METER _____ OTHER _____

PHYSICAL DESCRIPTION:

Layers			Physical			Color/Description			Clarity			Solubility		Reaction	
P	I	L	S	S	G	Oil	Syrup	Viscous,	C	C	O	W	H	A	W
H	N	I	O	L	E	Watery	Paste	Chunks	L	L	P	A	E	I	A
A	C	Q	L	U	L	Gel	Spongy	Soap-like	E	O	A	T	X	R	T
S	H	U	I	D		Soft	Hard	Powder	A	U	Q	E	A		E
E	E	I	D	G		Crystal	Granular	Rubbery	R	D	U	R	N		R
	S	D		E						Y	E		E		
Top															
Middle															
Bottom															

FIELD SCREENING RESULTS:

Layers	pH	Chlorine Hot Wire	Flammable	Cyanide	Oxidizer	Chloride	Peroxide	Mercury	Sulfide	PCB
Top										
Middle										
Bottom										

ASSIGNED WASTE STREAM - BASED ON INITIAL RCRA HAZARD

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TEST COMPATIBILITY RESULTS:

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Prepared by: _____

Date: _____

Drum Inventory Log Sheet

Specific Location	Type Of Container	Volume/ Capacity	Markings/ Identification/ Labels	Material in Container	Disposition Date	Disposition Place	Comments

ATTACHMENT D:

PCB Field Screening Procedure - SW 846 Method 9078

METHOD 9078

SCREENING TEST METHOD FOR POLYCHLORINATED BIPHENYLS IN SOIL

1.0 SCOPE AND APPLICATION

1.1 The method may be used to determine the amount of PCB (polychlorinated biphenyl) contamination in soils such as sand, gravel, loam, sediment, and clay, assuming that PCBs are the sole source of organic halogens in the sample.

1.2 This electrochemical method is designed to provide quantitative field results over a range of 2 to 2000 µg/g PCBs, significantly cutting down on the number of samples requiring laboratory testing.

1.3 Chlorines are removed from the PCB molecule using an organo-sodium reagent. The resulting chloride ions are measured using a chloride specific electrode. Analysts must identify the type of Aroclor contamination in order to use this as a quantitative method.

1.4 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

A sample of the soil to be tested is extracted with a hydrocarbon based solvent. The resulting extract is filtered to remove moisture and inorganic salts. The dried extract is reacted with metallic sodium and a catalyst to strip chloride from any PCB that may be present. The resulting chloride ions are extracted into an aqueous buffer solution where they are detected using a chloride ion specific electrode.

CAUTION: Some of the reagents used with this testing procedure contain flammable solvents, dilute acids, and metallic sodium. Wear gloves and safety glasses while performing tests. Read all MSDS and warnings included with the instrument before starting testing procedure.

3.0 INTERFERENCES

3.1 This procedure is sensitive to any chlorinated compound that is preferentially soluble in a non-polar solvent. When analyzing for PCBs, the presence of other chlorinated organics will result in a high bias. Iodine and bromine containing compounds will affect results if present in significant quantities. Wet or dry samples may be run, but results for all samples are calculated on a wet-weight basis. In one evaluation study (Table 1), 1.4% of the measurements were false negatives.

3.2 Inorganic chlorides should not interfere using this method if the sample is extracted with organic solvent.

4.0 APPARATUS AND MATERIALS

Electrochemical PCB test kit: L2000® PCB/Chloride Analyzer, (Dexsil Corporation, One Hamden Park Drive, Hamden, CT), or equivalent. Each commercially available test kit will supply or specify the apparatus and materials necessary for successful completion of the test.

5.0 REAGENTS

Each commercially available test kit will supply or specify the reagents necessary for successful completion of the test. Reagents should be labeled with appropriate expiration dates.

6.0 SAMPLE COLLECTION AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Sec. 4.1.

6.2 Soil samples may be contaminated, and should therefore be considered hazardous and handled accordingly. All samples should be collected using a sampling plan that addresses the considerations discussed in Chapter Nine.

6.3 To achieve accurate analyses, soil samples should be well homogenized prior to testing. PCBs are generally not evenly distributed in a soil sample and extensive mixing must be done to assure consistency.

7.0 PROCEDURE

Follow the manufacturer's instructions for the test kit being used. Those test kits used must meet or exceed the performance specifications indicated in Tables 1 and 2.

8.0 QUALITY CONTROL

8.1 Follow the manufacturer's instructions for quality control procedures specific to the test kit used. Additionally, guidance provided in Chapter One should be followed.

8.2 Use of replicate analyses, particularly when results indicate concentrations near the action level, is recommended to refine information gathered with the kit.

8.3 Method 9078 is intended for field or laboratory use. The appropriate level of quality assurance should accompany the application of this method to document data quality.

9.0 METHOD PERFORMANCE

9.1 146 soil samples from a PCB contaminated site were analyzed. There were 114 individual samples and 32 field duplicates. Each sample was analyzed using both the L2000 and GC/MS. The L2000 analyses were performed on-site in a mobile lab and the PCBs were analyzed as Aroclor 1242. Laboratory analyses were performed on splits of the same samples. The results from the analyses are presented in Table 1.

9.2 After applying accepted statistical methods to account for the detection limit difference between the two methods the data were evaluated to determine the acceptability of the L2000 method. A matched-pair students t-test performed on the L2000 and CLP GC/MS data results in a t value of 0.2141. This is well below the critical value (1.645 @ 0.05) for rejecting the null hypothesis indicating that there is no statistical difference between the data pairs. An analysis of the data for outliers identified only 2 data points whose residuals were greater than 3 standard deviations (10 and 5 respectively). Both points were determined to be in error using other evidence and were eliminated from the data set. A linear regression analysis of the remaining data results in a correlation coefficient of 0.95 and a positive intercept of 10.98 $\mu\text{g/g}$. The slope of 0.985 was not statistically different from 1 and the intercept was not statistically different from 0.

9.3 The relative percent difference (RPD) calculated from all valid duplicates greater than the L2000 detection limit of 2 $\mu\text{g/g}$ for each method resulted in a mean RPD of 19% for the L2000 data and a mean RPD of 43% for the CLP GC-MS method. A Dunnett's test shows that this is statistically significant.

9.4 In a second study, soil samples contaminated with Aroclor 1260 were taken during a site cleanup. The samples were split and sent for lab analysis by Method 8082 as well as analysis by the L2000 in the field. The results are reported in Table 2. A linear regression analysis of the data resulted in a correlation coefficient of 0.995, a slope of 1.048 and an intercept of -1.48 $\mu\text{g/g}$ indicating that the L2000 is accurate compared to the lab method. A calculation of the relative percent difference for data, where duplicates were run within a method, results in a lower RPD for the L2000 indicating a tighter data spread and better repeatability.

10.0 REFERENCE

1. Griffin, Roger D. Application of a New PCB Field Analysis Technique for Site Assessment. Proceedings of Hazmacon '92 March - April 1992.

TABLE 1
COMPARISON OF L2000 AND GC/MS RESULTS FROM SPLIT SAMPLES
Summary of Results

Sample Number	L2000 (µg/g)	GC/MS (µg/g)	Results Agree?
1	ND	0.593	Yes
3	ND	0.114	Yes
4	23.6	6.71	Yes
6	ND	0.679	Yes
7	ND	0.552	Yes
8	3.9	2	Yes
9	6.9	1.3	Yes
10	5.1	0.172	Yes
11	2.7	1.15	Yes
15	9.4	9.13	Yes
15D	12.5	9.84	Yes
16	484	2110	Yes
17	6.5	2.55	Yes
18	382	45.4	Yes
19	71.1	6.7	False Pos.
23	48.8	20.8	Yes
25	3.5	11.7	Yes
32	36	47.6	Yes
33	ND	6	Yes
34	14.4	34	Yes
36	>2000	816	Yes
38	778	1030	Yes
40	5.7	4.25	Yes
43	4.1	1.69	Yes
43D	3.6	1.74	Yes
50	ND	3.6	Yes
50D	ND	4.4	Yes
52	9.3	4.21	Yes
53	25.7	0.958	False Pos.
54	5.1	0.516	Yes
55	4.4	2.4	Yes
59	ND	7.9	Yes
60	2.3	0.624	Yes
60D	4.4	0.577	Yes
61	549	580	Yes

TABLE 1 (cont.)

Sample Number	L2000 (µg/g)	GC/MS (µg/g)	Results Agree?
62	111	2.35	False Pos.
64	172	19	Yes
65	ND	3.1	Yes
66	2.1	1.98	Yes
67	7.5	0.081	Yes
68	8	0.504	Yes
69	5.8	ND	Yes
69D	4.4	ND	Yes
73	37	15.8	Yes
74	22	13.3	Yes
75	61	23	Yes
76	82	46.7	Yes
78	21	2.27	Yes
79	148	42.8	Yes
80	ND	3.8	Yes
84	7.6	1.16	Yes
84D	10.9	1.08	False Pos.
85	593	428	Yes
85D	596	465	Yes
88	ND	2.7	Yes
88D	ND	1.77	Yes
89	ND	45	False Neg.
90	2	1.01	Yes
90D	ND	1.4	Yes
91	1650	1630	Yes
91D	1608	1704	Yes
92	3.14	1.21	Yes
92D	3.4	ND	Yes
95	20.6	17.5	Yes
95D	20.1	31.2	Yes
100	384	177	Yes
100D	363	167	Yes
101	8.3	1.21	Yes
102	6.3	293	False Neg.
102D	5	1.77	Yes
103	75.2	40.3	Yes
104	4.1	7.66	Yes

TABLE 1 (cont.)

Sample Number	L2000 ($\mu\text{g/g}$)	GC/MS ($\mu\text{g/g}$)	Results Agree?
107	161	14.1	Yes
108	6.1	3.84	Yes
109	P	ND	Yes
109D	10.3	ND	False Pos.
111	20	ND	False Pos.
112	240	315	Yes
113	21.8	14.9	Yes
114	107	66.3	Yes

NOTE: 75 out of 146 samples are reported in Table 1. Samples that were found to be ND for both the L2000 kit and the GC/MS determination were not reported. The determination of a "false negative" result for the L2000 technique is based on an action level of 10 $\mu\text{g/g}$. If another action limit is chosen, the rate of false negative results may differ. Similarly, a "false positive" result for the L2000 technique is indicated when the L2000 results are above 10 $\mu\text{g/g}$ and the GC/MS results are "ND" or below 10 $\mu\text{g/g}$, or when the results of the L2000 techniques are higher than the GC/MS results by more than two orders of magnitude.

ND = Not detected

6 False positives: ND - 14.1 ppm by GC/MS

2 False negatives: 2.7 - 293 ppm by GC/MS

71 Non-detects: ND - 2.5 ppm by GC/MS

TABLE 2
COMPARISON OF L2000 AND GC/EC RESULTS FROM SPLIT SAMPLES
Summary of Results

Sample Number	Method 8082 (µg/g)	L2000 Results (µg/g)
1	83	79/76
2	21	22
3	12	14
4	300/375	357/326/327
5	29	27
6	106/134	116/117
7	3	7.6
8	9.3	7.2
9	1.5	5.2
10	99	93
11	7/9	13
12	3.6	12
13	4.2/6.2	2.9
14	290	254/265